Omega Chemical Superfund Site

Sampling and Analysis Plan Addendum for Additional Data Collection in the Phase 1a Area

~~May 31~~September 6, 2002

Submitted to:

U.S. Environmental Protection Agency
Region 9, Superfund Division
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September 6, 2002

Ms. Nancy Riveland-Har
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Subject: Submittal of Revised Draft (Redline-Strikeout) SAP Addendum
Omega Chemical Superfund Site
CDM Project No. 10500-30697-TO2.SAP
CDM File No. 10500-5.2.3

Dear Ms. Riveland-Har:

On behalf of the Omega Chemical Site PRP Organized Group (OPOG), Camp Dresser & McKee Inc. (CDM) is herein submitting two copies of the revised draft (redline-strikeout) SAP Addendum for Additional Data Collection in the Phase 1a Area (CDM, September 6, 2002) , for your review. The revised document incorporates OPOG responses to USEPA comments received in correspondence dated July 29, 2002. One copy has also been transmitted to Carol Yuge for her review.

Please feel free to contact me or Chuck McLaughlin (909/222-0387) if you have any questions.

Sincerely,

CAMP DRESSER & MCKEE INC.

Sharon Wallin

Sharon L. Wallin, R.G.
Project Manager

Enclosure

cc: Carol Yuge, Roy F. Weston, Inc.
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Section 1
Introduction, Background, and Recent Data

1.1 Introduction

On behalf of the Omega Chemical Site PRP Organized Group (OPOG), Camp Dresser & McKee Inc. (CDM) has prepared this Sampling and Analysis Plan (SAP) Addendum for Additional Data Collection in the Phase 1a Area. This SAP Addendum has been prepared and the proposed work will be performed in partial fulfillment of the Statement of Work specified in Consent Decree No. 00-12471 between USEPA and OPOG lodged on November 24, 2000 and entered into the U.S. District Court on February 28, 2001.

The Phase 1a Area is defined in the Consent Decree as "the area of soil and groundwater contamination associated with the Omega Property and extending downgradient approximately 100 feet southwest of Putnam Street, Whittier, California". The locations of the Omega Property (Site) and Phase 1a Area are illustrated on Figure 1-1.

1.2 Background

Task 1 of the Consent Decree states that OPOG will design and implement a groundwater containment and mass removal treatment system in the Phase 1a Area. The purpose of the proposed investigation is to collect additional data (e.g., lithologic, water quality, aquifer hydraulics, etc.) in order to assist in the selection, design, and implementation of the groundwater remedy in the Phase 1a Area, and complete the Engineering Evaluation/ Cost Analysis (EE/CA) Report. The additional data collection activities proposed in this SAP Addendum will assist with an evaluation of potential extraction well locations.

This document has been developed as an Addendum to the USEPA-approved Downgradient Well Installation and Groundwater Monitoring SAP (CDM, April 20, 2001). At the time the Downgradient Well SAP was prepared, OPOG had installed and sampled four groundwater monitoring wells (OW-1, OW-1b, OW-2, and OW-3). With the approval of the Downgradient Well SAP, OPOG installed and sampled four additional downgradient wells (OW-4a, OW-4b, OW-5, and OW-6). Three of the downgradient wells were installed during March and April 2001 (OW-4a, OW-4b, and OW-6), with the fourth well (OW-5) installed in August following evaluation of the initial water quality sampling results for wells OW-4a and OW-4b. The locations of all Omega wells are illustrated on Figure 1-2.

The Downgradient Well SAP also specified the collection of four quarterly sampling events and monthly water levels for one year from all Omega wells. Groundwater samples, therefore, were collected from the wells during mid-May, mid-August, and mid-November 2001 and mid-February 2002. Quarterly sampling results have been entered into the project's Access database and are tabulated and discussed in Section 1.3 of this document.

In mid-2001, USEPA requested that OPOG install and sample an additional groundwater monitoring well upgradient of the site. Following discussions between USEPA staff and OPOG Steering and Technical Committee members, OPOG agreed to install and sample the upgradient well. Additional data requirements for the Omega Groundwater Remedy were also discussed in a Technical Memorandum from OPOG to USEPA dated October 31, 2001. The Technical Memorandum identified the following additional data requirements:

- Installation of a third monitoring well (OW-8) at Putnam Street, between OW-2 and OW-3, to verify the lateral distribution of VOCs at this location and to collect additional hydrostratigraphic and hydraulic data at this location;
- Single well aquifer recovery tests at wells OW-2, OW-3, OW-4a and the new Putnam Street well location (OW-8) to provide better estimates of hydraulic conductivity at these locations; and
- Addition of 1,4-dioxane, metals, bioparameters (e.g., electron donors and receptors), TDS, TOC, and COD to the analytical suite for the next round of sampling at OW-1, OW-1b, OW-2, OW-3, OW-4a and OW-4b.

Access was obtained during late-2001 and early-2002 from Caltrans for upgradient well OW-7 and from the City of Whittier for Putnam Street well OW-8. In order to minimize drilling mobilization costs, wells OW-7 and OW-8 were installed concurrently in mid-March 2002. The wells were sampled on March 27, 2002, seven days following the completion of well development. Well drilling, installation, development, and sampling activities were performed in accordance with the procedures specified in the Downgradient Well SAP.

The additional data requirements listed above form the basis of the activities proposed in this SAP. Where applicable, procedures detailed in the Downgradient Well SAP will be referenced in this SAP. Procedures not included in the Downgradient Well SAP will be detailed in Sections 2 and 3 of this document. As discussed above, the additional Putnam Street well has already been installed, therefore, well drilling, installation, and development procedures are not discussed in this document.

1.3 Recent Data

During 2001 and 2002, additional data were obtained which increased our knowledge of subsurface conditions in the Phase 1a Area. Provided below is a brief discussion of the recently-acquired data (i.e., water quality data, water level and groundwater elevation data, lithologic and well development data). OPOG initiated semi-annual sampling of all 10 Omega wells during mid-August 2002. The analytical results were pending at the time this document was prepared.

1.3.1 Water Quality Data

As discussed previously, water quality samples were collected quarterly from all Omega wells for one year starting mid-May 2001 and ending mid-February 2002.

These results including historical results are summarized in Tables 1-1 through 1-5. In addition, analytical results for new wells OW-7 and OW-8 sampled in late-March 2002 are also included in the summary tables. Chlorinated VOC concentrations in on-site well OW-1 remained significantly elevated, however, tetrachloroethene (PCE) concentrations were observed to decline from 86,000 to 30,000 micrograms per liter [µg/L]) during the period from May 2001 to February 2002. Chlorinated VOCs in near-site deeper well OW-1b were generally not detected, with the exception of tetrachloroethene (PCE) which ranged from 29 to 62 micrograms per liter (µg/L), and low levels of trichloroethene (TCE) and 1,2-dichloroethane (1,2-DCA). Chlorinated VOCs in downgradient Putnam Street wells OW-2 and OW-3 were also detected at elevated concentrations, however, the concentrations were significantly less than those observed in the on-site well (e.g., PCE ranged from 620 to 780 µg/L in OW-2, and from 2,100 to 1,400 µg/L in OW-3.

Concentrations of chlorinated VOCs detected in Washington Boulevard well OW-4a were also elevated and comparable to detections in OW-2 and OW-3, however, VOCs were generally not detected in the deeper well at that location (OW-4b). The exception was PCE, which was detected at concentrations ranging from 1.2 to 1.9 µg/L, below the maximum contaminant level (MCL) of 5 µg/L.

The initial sample from upgradient well OW-7 indicated that chlorinated VOCs were detected at that location. PCE was detected at a concentration of 5.6 µg/L, in addition Freon 113 and Freon 11 were also detected at concentrations of 62 and 36 µg/L, respectively. The initial sample collected from additional Putnam Street well OW-8 indicated that a variety of chlorinated VOCs were present at elevated concentrations. The compounds detected were similar to those detected at the location of on-site well OW-1. Subsequent sampling of these new wells will allow for a determination to be made whether these detections were a relict of cross-contamination during drilling or are present and persistent.

The emerging compound 1,4-dioxane was analyzed at locations OW-1 through OW-6 during November 2001 and February 2002. Elevated concentrations of 11,000 and 41 µg/L were detected at locations OW-1 and OW-1b, respectively.

Semi-VOCs and pesticides were analyzed for and not detected at locations OW-1 and OW-1b during all four quarterly sampling events. Total and dissolved metals were also analyzed at these two locations and were generally found at background concentrations during all four quarterly sampling events. Perchlorate was also analyzed and not detected during all four quarterly sampling events.

1.3.2 Water Level and Groundwater Elevation Data

Depth-to-water measurements were collected from all Omega wells monthly for one year, beginning with the May 2001 sampling event. All Omega wells were surveyed and groundwater elevation calculated at each location. Groundwater elevation results are summarized in Table 1-6. As indicated on the groundwater elevation contour maps (Figures 1-3 through 1-14), the direction of groundwater flow was

consistently towards the southwest during all 12 months. There is a noticeable change in hydraulic gradient in the vicinity of Washington Boulevard, which corresponds to the observed transition from finer-grained subsurface lithology in the area northeast of Washington Boulevard to coarser-grained subsurface lithology in the area southwest of Washington Boulevard.

As observed at the two locations where shallow and deeper well pairs (OW-1 and OW-4) are present, groundwater elevations in the deeper wells generally ranged from 4 to 7 feet deeper than the elevations observed at the shallow wells at those locations. This head difference suggests that some degree of hydraulic separation exists between the shallow and deeper screened zones. The head differences also indicate a downward hydraulic gradient at these locations, suggesting that there is the potential for contaminants to migrate downward towards the deeper zone. Water quality results from the two well pair locations support the assumption that hydraulic separation between the two zones limits downward vertical migration.

1.3.3 Lithologic Data

The subsurface lithology at the location of new well OW-7 was very similar to the lithology at locations OW-1 and OW-1b. As indicated on the lithologic logs contained in Appendix A, the subsurface materials at location OW-7 consisted primarily of clays and silty clays. The subsurface materials at location OW-8 were comparable to the materials observed at locations OW-2 and OW-3, with silts and clays observed above 54 feet below ground surface (bgs) and sand observed in the interval from 54 to 79 feet bgs. At location OW-4, the subsurface materials were coarser-grained, and consisted of sands and silty sands interbedded with clays and silty clays. Due to flowing sands encountered at location OW-4, the deeper well (OW-4b) was drilled using the mud rotary drilling method. An electric log was performed in the boring and is included in Appendix A.

1.3.4 Well Development Data

Well development logs for the four new wells installed in the vicinity of the Phase 1a Area (OW-4a, OW-4b, OW-7 and OW-8) are contained in Appendix B. As indicated on the logs, upgradient well OW-7 was not capable of sustaining a pumping rate in excess of ~~one~~ two gallons per minute (gpm), which was comparable to that observed at locations OW-1 and OW-1b. Wells OW-4a, OW-4b, and OW-~~7~~8 were capable of sustaining rates of 5 gpm or slightly greater (up to 10 gpm). Wells OW1, OW-1b, and OW-7, therefore, are not suitable for test pumping purposes, whereas new wells OW4a, OW-4b and OW-~~7~~8 and existing wells OW-2 and OW-3 are capable of ~~sustained pumping and may be test pumped~~sustaining a minimum pumping rate of one gpm. The remaining wells (OW-4b, OW-5 and OW-6) are either completed in a deeper portion of the shallow zone or are located too far downgradient from the Phase 1a area to be considered for potential extraction locations.

Section 2
Field Sampling Procedures

As discussed in Section 1, two tasks will be performed during the investigation proposed in this SAP Addendum:

- Task 1 - Aquifer Testing and Water Quality Sampling and Analysis
- Task 2 - Semi-Annual Monitoring Well Sampling and Analysis

Included in Appendix B of the Downgradient Well SAP are the following field forms that will be utilized during the performance of the field activities proposed herein:

- Monitoring Well Purge and Sampling Form
- Chain-of-Custody Record
- Example Sample Label

The following form is not contained in the Downgradient Well SAP and so is included in Appendix B of this SAP Addendum:

- Aquifer Pump Test Form

As previously discussed, this SAP Addendum references the Downgradient Well SAP where applicable. Sufficient detail has been provided in this section to allow field personnel to conduct the proposed field activities. However, for general guidance and reference purposes, Standard Operating Procedures (SOPs) for applicable field activities have been included in Appendix C of the Downgradient Well SAP. The SOPs have been previously reviewed and approved for similar site activities by either USEPA Regions VIII or IX, or are modifications of SOPs that have been approved by USEPA.

The following SOPs are included in Appendix C of the Downgradient Well SAP and are applicable to this investigation:

- Groundwater Sampling
- Field Logbook
- Chain-of-Custody Procedures

Equipment calibration and operating procedures for the following field equipment are included in Appendix D of the Downgradient Well SAP:

- Thermo Environmental 580B Photoionization Detector
- YSI Model 33 Conductivity Meter
- LaMotte 2008 Turbidity Meter

The following SOP is not contained in the Downgradient Well SAP and so is included in Appendix D of this SAP Addendum:

- Aquifer Hydraulic Tests

2.1 Task 1 - Aquifer Testing and Water Quality Sampling and Analysis

As discussed previously in Section 1.2, up to four wells (OW-2, OW-3, OW-4a and OW-8) will be test pumped in order to estimate aquifer parameters at the tested well locations. All field work will be performed in accordance with the Health and Safety Plan developed for the prior Phase 1a field investigation (CDM, December 15, 1998).

2.1.1 Aquifer Testing

Constant discharge pumping will be performed for an approximate 4-hour period, with recovery measured until the water level has recovered to within 95% of its pre-test static condition.

Based on observations made during well development, the following pumping rates have been assumed for the proposed constant discharge testing:

- OW2 - 5 gpm
- OW3 - 1 gpm
- OW8 - 7 gpm
- OW4a - 10 gpm

Approximately 5,500 gallons of purge water will be generated during the four tests. The water will be temporarily contained in a portable tank on site. Upon the completion of testing, a composite sample will be collected for pre-disposal VOC analysis. Pending evaluation of the pre-disposal sample results, the purge water will be transported to a recycling facility for disposal.

Water levels prior to initiating each test and during the pumping and recovery phases of each test will be monitored automatically using a data logger and transducer, and manually using an electric water level indicator. Depending on equipment availability, data loggers and transducers manufactured by In-Situ (e.g., Hermit or Trolls) will be utilized to collect water level measurements during testing. Equipment operation will be performed in accordance with manufacturer's instruction manuals. Because equipment availability is unknown at present, manuals will be provided in the field for review prior to the initiation of aquifer testing.

Manual water level readings will be collected on a typical logarithmic progression (e.g., every minute during the first ten minutes of the test, every two minutes from 10 to 20 minutes into the test, every 5 minutes from 20 to 30 minutes into the test, every 10 minutes from 30 to 60 minutes into the test, etc.). The data logger will also collect water level measurements using its pre-set logarithmic progression.

The pumping rate will be measured using an in-line flowmeter and totalizer. The volume of water pumped during each test and the time pumped will be noted. Periodically during pumping, samples of the discharge water will be collected for field measurement of pH, temperature, electrical conductivity, and turbidity. Samples will also be collected for laboratory analysis prior to termination of pumping, as described below. Upon the completion of testing at all wells, the data will be analyzed to provide estimates of hydraulic conductivity at the tested locations.

Aquifer testing and data evaluation will be performed in general accordance with the Aquifer Hydraulic Tests SOP contained in Appendix D of this document. Data evaluation methods may be revised pending analysis. Equipment decontamination will be performed as described in Section 4.8 of the Downgradient Well SAP.

2.1.2 Water Quality Sampling and Analysis

Water quality samples will be collected from each pumped well just before the termination of constant discharge pumping. One sample will be collected from each tested well and submitted for analysis of the following parameters on a standard turnaround basis:

- VOCs plus acetone, Freon 11, Freon 12, Freon 113, MTBE (methyl-tertiary-butyl-ether) and Tentatively Identified Compounds (TICs) by Method 8260B
- 1,4-Dioxane by Method 8270M

The discharge rate will be slowed to less than one gpm during sample collection. The sample containers will be filled directly from the end of the discharge pipe or a sampling tap located on the discharge line. One sequential sample will also be collected from well OW-8 at the time of sampling. Sample collection and handling will be performed in accordance with the procedures specified in Section 2.1.5 of the Downgradient Well SAP. Proposed samples and analytical parameters are summarized in Table 2-1. Sample collection and handling procedures will be as described in Section 2.2 of this document.

2.2 Semi-Annual Monitoring Well Sampling and Analysis

Starting with the completion of quarterly sampling in February 2002, all 10 Omega wells will be sampled on a semi-annual basis until the Phase 1a Area treatment plant is operational. As previously discussed, ~~Semi-~~semi-annual sampling~~, therefore, will be~~ was initiated during mid-August 2002. All 10 wells will be analyzed for the following parameters at a fixed-base laboratory during all subsequent semi-annual sampling events:

- VOCs plus acetone, Freon 11, Freon 12, Freon 113, MTBE (methyl-tertiary-butyl-ether) and Tentatively Identified Compounds (TICs) by Method 8260B

In addition, wells OW-1, OW-1b, OW-2, OW-3, OW-4a, OW-4b, and OW-8 were analyzed for 1,4-dioxane during the mid-August 2002 sampling event. These wells will also be sampled for 1,4-dioxane during the February 2003 sampling event.

As indicated in Section 2.1.5 of the Downgradient Well SAP, the following field parameters will be measured during sampling:

- Specific conductance and temperature
- pH
- turbidity

As indicated in the OPOG Technical Memorandum dated October 31, 2001, the following additional analyses for biodegradation/natural attenuation parameters and emerging compounds will also be performed on groundwater samples collected from wells OW-1, OW-1b, OW-2, OW-3, OW-4a, OW-4b and OW-8 during the ~~next~~ February 2003 semi-annual sampling event. Analyses of the field parameters listed below will be performed in accordance with manufacturer's directions provided with each Direct Reading Instrument (DRI) and Hach Test Kit (see Appendix C for instruction manuals).

Biodegradation/Natural Attenuation Field Parameters

The following biodegradation/natural attenuation parameters will be analyzed immediately in the field:

- Dissolved Oxygen *
- Redox (Eh) *
- Sulfate **
- Iron (II) ** (field filtering will be performed if turbidity >50 NTUs)
- Alkalinity **
- Chloride **
- Hydrogen Sulfide **
- Carbon Dioxide **

* Indicates field analysis using a DRI (manufacturer's instruction manual for the Orion 250A which measures pH, Eh, and DO is included in Appendix C).

** Indicates field analysis performed per manufacturer's instructions using a Hach Test Kit.

Biodegradation/Natural Attenuation Analytical Parameters (fixed-base laboratory)

- Nitrate/Nitrite

- Dissolved Organic Carbon (indicate on COC "filtering required by the lab")
- Methane/Ethane/Ethene (if field tests indicate conditions are anaerobic)

Sample handling will be performed as indicated in Section 3 (Table 3-1). It should be noted that laboratory samples for dissolved organic carbon analysis will be collected in unacidified containers. The Chain of Custody (COC) form will indicate that the sample requires filtering and acidification upon receipt by the analytical laboratory. In addition, field personnel will coordinate with the analytical laboratory to make sure that analyses with short holding times (e.g., nitrate/nitrite and hexavalent chromium) are analyzed within the required holding time.

Bottles will be filled for methane/ethane/~~ethane~~ ethene analyses and stored in an iced cooler pending evaluation of the dissolved oxygen measurements and ferrous iron (Fe II) results for each well sampled. In the event that anaerobic conditions are observed at a sampled well location (e.g., ferrous iron is detected during field testing and dissolved oxygen measurements are less than 1 mg/L), the bottles filled for methane/ethane/ ~~ethane~~ ethene analyses will be submitted to the fixed-base laboratory for analysis. Methane/ethane/ethane are metabolic byproducts produced only under reduced, anaerobic environments. Methane is produced through carbon dioxide reduction and/or fermentation reactions, while ethane and ethane are innocuous end-products that result from the reductive declorination of chlorinated VOCs. In the absence of anaerobic conditions, methane/ethane/ethene generation will likely be insignificant, therefore, analysis for these compounds in the event that anaerobic conditions are observed, is unwarranted.

Emerging Compounds (fixed-base laboratory)

- Hexavalent Chromium
- 1,4-Dioxane
- Perchlorate

Because the above parameters were not included in the Downgradient Well SAP, testing methods and bottle requirements are summarized in Section 3 (see Table 3-1) of this SAP Addendum.

Each well will be purged using a portable submersible pump and dedicated polyethylene tubing which has been installed inside each Omega well. Upon the completion of purging, with the exception of samples for VOC and 1,4-dioxane analysis, all sample containers will be filled directly from the end of the discharge tubing. The discharge rate will be lowered to less than one gpm during filling of the sample containers. Upon the completion of sample collection, the submersible pump will be removed from the well and a precleaned, disposable bailer lowered to the approximate middle of the perforated section. The bailer will be used to collect samples for VOCs and 1,4-dioxane analyses. The sample will be poured directly into the sample containers, minimizing agitation. After sampling is completed, the bailer and line will be discarded.

Samples will be submitted for standard analytical turnaround time (approximately two weeks). Level 4 deliverables will be requested on 10% of the samples submitted for fixed-base laboratory analysis during each sampling event, in order to perform data validation. The laboratory will provide both electronic and hard copy reports. Analytical summary tables will be provided to USEPA for review approximately two to three weeks following receipt of the final analytical results from the laboratory.

Water level measurements will also be collected from all Omega wells prior to each sampling event. As previously discussed, water level measurements and water quality sampling activities will be performed in accordance with the procedures specified in Sections 2.1.4 and 2.1.5 of the Downgradient Well SAP, respectively.

TABLE 2-1
PROPOSED SAMPLE SUMMARY

Task	Description	Fixed-Base Laboratory Analyses							Field Analyses	
		VOCs [3] (8260B)	1,4-Dioxane (8270M)	Hex. Cr. (7199)	Perchlorate (314)	NO_2/NO_3 (300)	DOC (9060)	M/E/E [5] (AM20)	Hach Test Kits [6]	DO/ Redox [7]
2.1	**Aquifer Testing**									
	Groundwater [1]	4	4							
	Duplicate	1	1							
	Subtotal	5	5							
2.2	**Monitoring Well Sampling**									
	Groundwater [2,4,8]	10	7	7	7	7	7	7	7	7
	Duplicate	1	1	1	1	1	1	1	1	1
	Equipment Blank	1	1	1	1	1	1	1		
	Subtotal	12	9	9	9	9	9	9	8	8
	Total Samples	17	14	9	9	9	9	9	8	8

1 Wells OW2, OW3, OW4a, OW8

2 Wells OW1, OW1b, OW2, OW3, OW4a, OW4b, OW5, OW6, OW7 and OW8 for VOC analysis

3 VOCs plus Acetone, Freon 11, Freon 12, Freon 113, MTBE and TICs; Level IV deliverables requested on 10% of the samples

4 1,4-Dioxane, Hex. Cr., Perchlorate, NO_2/NO_3, and DOC (M/E/E, if required) on selected wells (OW1, OW1b, OW2, OW3, OW4a, OW4b, OW8)

5 M/E/E analysis only if field tests indicate conditions are anaerobic (e.g., ferrous iron is detected in the sample and DO measurement < 1mg/l)

6 Field Analyses for Biodegradation/Natural Attenuation Parameters (sulfate, ferrous iron, alkalinity, chloride, hydrogen sulfide and carbon dioxide)

7 Field Analyses for Biodegradation/Natural Attenuation Parameters using Direct Reading Instruments (DRI)

8 One sample with assumed low concentrations will be selected for laboratory MS/MSD (triple volume) for all analytes except M/E/E

Note: Analyses indicated above will be performed during the February 2003 sampling event. During all subsequent semi-annual sampling events, only VOCs (plus Acetone, Freons, MTBE and TICs) will be analyzed.

M/E/E - Methane, Ethane, Ethene
DO - Dissolved Oxygen
TICs - tentatively identified compounds
NO_2/NO_3 - Nitrite/Nitrate
VOCs - Volatile Organic Compounds
MTBE - methyl-tertiary-butyl-ether
Hex. Cr. - Hexavalent Chromium
DOC - Dissolved Organic Carbon

Section 3
Quality Assurance/Quality Control Procedures

Quality Assurance/Quality Control (QA/QC) procedures will be performed as described in Section 4 of the Downgradient Well SAP. As described in Section 2 of this SAP Addendum, analyses for biodegradation/natural attenuation parameters and emerging compounds were not included in the Downgradient Well SAP. Therefore, a summary table of sampling requirements and analytical methods for these parameters and methods has been prepared and included in this section (see Table 3-1). In addition, quantitation, precision and accuracy limits for these parameters and analytical methods has been summarized in Table 3-2.

Table 3-1
Biodegradation/Natural Attenuation Parameters and Emerging Compounds
Sampling Requirements and Analytical Methods

	Recommended Instrument	Analytical Method	Bottle Req.	Field Filtered (Y/N)	Preservative
Field Parameters					
Eh	*Orion 250A*	DRI	N/A	N	N/A
Dissolved Oxygen	*Orion 250A*	DRI	N/A	N	N/A
pH	*Orion 250A*	DRI	N/A	N	N/A
Specific Conductivity	*YSI Model 33*	DRI	N/A	N	N/A
Turbidity	*LaMotte 2008*	DRI	N/A	N	N/A
Temperature	*Orion 250A*	DRI	N/A	N	N/A
Ionic Parameters (On-site Analyses)					
Sulfate	HACH (DR2000 or Similar Colormetric)	HACH 8051	1-500 mL plastic bottle	N	Cool 4°C
Iron (II)		HACH 8146		Y, if necessary [2]	Cool 4°C
Alkalinity		HACH 8221		N	Cool 4°C
Chloride		HACH 8113		N	Cool 4°C
Hydrogen Sulfide	HACH Kit	HACH 8131	Supplied w/ kit	N	Cool 4°C
Carbon Dioxide	HACH Kit	HACH 8205 or 8223	Supplied w/kit	N	Cool 4°C
Organics and Inorganics (Fixed-Base Laboratory)					
Nitrate/Nitrite	—	IC Method E 300	1-L Polyethylene	N	H_2SO_4 Cool 4°C
Methane/Ethane/ Ethene[1]	—	AM20	2-40 ml VOA vials	N	None
Dissolved Organic Carbon	—	EPA 9060	1-125 ml amber glass	N [3]	Cool 4°C
Hexavalent Chromium	—	EPA 7199	1-500 ml Polyethylene	Y	NAOH Cool 4°C
1,4-Dioxane	—	EPA 8270M	1-1L amber glass	N	Cool 4°C
Perchlorate	—	EPA 314	1-500 ml Polyethylene	N	Cool 4°C

N/A – Not applicable
DRI = direct reading instrument
[1] The recommended analytical method is AM20 (Microseeps Analytical, Pittsburgh, PA).
[2] Field filtering will be performed using a 0.45 micron filter in the event that turbidity measurements exceed 50 NTUs.
[3] The Chain of Custody form will indicate that the laboratory must filter and preserve the sample with H_2SO_4 upon receipt.

TABLE 3-2
QUANTITATION, ACCURACY AND PRECISION LIMITS
Biodegradation/Natural Attenuation and Emerging Compounds Analyses

	Aqueous Samples				
			MS/MSD		*LCS*
Analyte	*Analytical Method*	*Laboratory Quantitation Limit*	*Accuracy: Recovery (%)*	*Precision: RPD (%)*	*Recovery (%)*
Nitrate/Nitrite	EPA 300	0.15 mg/L	75 - 125%	20%	85 - 115%
Methane/ Ethane/ Ethene	AM20	15 ng/L 5 ng/L 5 ng/L	Determined by laboratory	Determined by laboratory	Determined by laboratory
Dissolved Organic Carbon	EPA 9060	1 mg/L	80 - 120%	20%	85 - 115%
Hexavalent Chromium	EPA 7199	0.5 µg/L	90 - 110%	20%	90 - 110%
1,4-Dioxane	EPA 8270M	3 µg/L	Determined by laboratory	Determined by laboratory	Determined by laboratory
Perchlorate	EPA 314	4 µg/L	80 - 120%	20%	85 - 115%

Notes:

LCS	Laboratory Control Sample
MS/MSD	Matrix Spike/Matrix Spike Duplicate
RPD	Relative Percent Difference
ng/L	Nanograms per liter
µg/L	Micrograms per liter
mg/L	Milligrams per liter

Appendix C
Orion 250A and Hach Test Kit Instruction Manuals

CARBON DIOXIDE TEST KIT
Model CA-23
Cat. No. 1436-01

HACH

Low Range

1. Fill the mixing bottle to the 23-mL mark with the water sample.
2. Add one drop of Phenolphthalein Indicator Solution to the sample.
3. Add the Sodium Hydroxide Solution drop by drop to the sample. Count each drop as it is added. Swirl the bottle to mix after each drop is added as shown in Figure 1. Continue adding drops until a light pink color forms and persists for 30 seconds.
4. Each drop of Sodium Hydroxide Solution used equals 1.25 mg/L carbon dioxide (CO_2).

WARNING: The chemicals in this kit may be hazardous to the health and safety of the user if inappropriately handled. Please read all warnings before performing the test and use appropriate safety equipment.

HACH COMPANY, P.O. BOX 389, LOVELAND, COLORADO 80359
TELEPHONE: WITHIN U.S. 800-227-4224, OUTSIDE U.S. 970-669-3050, TELEX: 160840

Medium Range

1. Fill the mixing bottle to the 15 mL mark with the water sample.
2. Add one drop of Phenolphthalein Indicator Solution to the sample.
3. Add the Sodium Hydroxide Solution drop by drop. Count each drop as it is added. Swirl the bottle to mix after each drop is added. Continue adding drops until a light pink color forms and persists for 30 seconds.
4. Each drop of Sodium Hydroxide Solution used equals 2 mg/L carbon dioxide (CO_2).

High Range

1. Fill the plastic measuring tube level full with the water to be tested. Transfer to the mixing bottle by placing the mixing bottle over the tube and then turning the bottle right-side up.
2. Add one drop of Phenolphthalein Indicator Solution to the contents of the mixing bottle.
3. Add the Sodium Hydroxide Solution drop by drop. Count each drop as it is added. Swirl the bottle to mix after each drop is added. Continue adding drops until a light pink color forms and persists for 30 seconds.
4. Each drop of Sodium Hydroxide Solution used equals 5 mg/L carbon dioxide (CO_2).

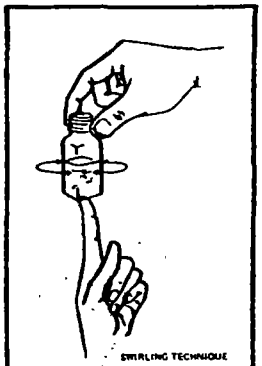


Figure 1

REPLACEMENTS

Cat. No.	Description	Unit
1897-36	Phenolphthalein Indicator Solution, 1 g/L	15 mL (½ oz) SCDB*
671-37	Sodium Hydroxide Solution, 0.01*N*	118 mL (4 oz) MDB†
438-00	Measuring Tube	each
2327-06	Mixing Bottle	pkg/6

*Self-contained Dropping Bottle
†Marked Dropping Bottle

24443-89

• Alkalinity Test Kit
• Trousse d'analyse alcalinité
• Alkalitäts-Test-Kit
• Kit para la determinación de alcalinidad

5–100, 20–400 mg/L
•Mod. AL-AP MG/L
•# 24443-01

- To ensure accurate results, read carefully before proceeding.
- Pour obtenir des résultats exacts, lire attentivement le mode d'emploi avant d'utiliser la trousse.
- Um genaue Ergebnisse zu gewährleisten, lesen Sie das Folgende bitte aufmerksam durch, bevor Sie fortfahren.
- Para obtener resultados precisos lea detenidamente las instrucciones antes de proceder al análisis.

WARNING

Handling chemical samples, standards, and reagents can be dangerous. Review the Material Safety Data Sheets before handling any chemicals.

ATTENTION

La manipulation des échantillons chimiques, étalons et réactifs peut être dangereuse. Lire les fiches de données de sécurité des produits avant de manipuler tout produit chimique.

WARNUNG

Die Handhabung chemischer Proben, Standards und Reagenzien kann gefährlich sein. Bitte gehen Sie die Materialsicherheitsdatenblätter durch, bevor Sie Chemikalien handhaben.

ADVERTENCIA

El manejo de sustancias químicas, patrones y reactivos, puede resultar peligroso. Lea las fichas de informaciones de seguridad de materiales antes de manipular cualquier producto químico.

HACH

Measuring Hints and General Test Information

- Wash all labware between tests. Contamination may alter test results. Clean with a non-abrasive detergent or a solvent such as isopropyl rubbing alcohol. Use a soft cloth for wiping or drying. Do not use paper towels or tissue on plastic tubes as this may scratch them. Rinse with clean water (preferably deionized water).
- When titrating, count each drop of titrant. Hold the dropper vertically. Swirl the mixing bottle after each drop is added.
- The result can be expressed in grains per gallon (gpg) by dividing the mg/L result by 17.1.
- To open PermaChem® Powder Pillows:
  1. Tap the bottom of the pillow on a hard surface.
  2. Tear open the pillow along the dashed line.
  3. Open the pillow and form a spout by squeezing the side edges.
  4. Pour the contents into the sample.
- Hach strongly recommends that, for optimum test results, reagent accuracy be checked with each new lot of reagents. Use the standard solution included in this kit or listed in the *OPTIONAL REAGENTS AND EQUIPMENT* section. Follow the instructions included with each standard solution.

Conseils pour les mesures et informations générales sur l'analyse

- Laver toute la verrerie entre les analyses. La contamination peut fausser les résultats d'analyses. Laver avec un détergent non abrasif ou un solvant tel que l'isopropanol. Utiliser un tissu doux pour essuyer ou sécher. Ne pas utiliser de tissu ou papier d'essuyage sur les tubes en plastique pour ne pas les rayer. Rincer à l'eau propre de préférence de l'eau désionisée.
- Lors du titrage, compter chaque goutte de titrant. tenir le compte-gouttes verticalement. Agiter le flacon carré après chaque goutte.
- Le résultat peut être exprimé en grains par gallon (gpg) en divisant le résultat en mg/L par 17.1, ou en degré français en divisant le résultat par 10.
- Pour ouvrir les sachets PermaChem®:
  1. Taper le bas du sachet sur une surface dure.
  2. Déchirer le sachet en suivant le pointillé.
  3. Ouvrir le sachet et former un bec en rapprochant les bords latéraux.
  4. Verser le contenu dans l'échantillon.
- Pour de meilleurs résultats, Hach recommande vivement de vérifier la validité du réactif pour chaque nouveau lot de réactifs. Utiliser la solution étalon contenue dans cette trousse ou listée dans la partie *REACTIFS ET EQUIPEMENTS OPTIONNELS*. Suivre les instructions fournies avec chaque solution étalon.

Meßtips und allgemeine Testinformationen

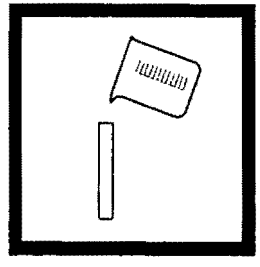
- Waschen Sie alle Laborartikel zwischen den Tests. Verunreinigung kann die Testergebnisse verfälschen. Reinigen Sie sie mit einem nicht scharfen Detergent oder einem Lösungsmittel wie zum Beispiel Isopropylalkohol. Verwenden Sie für das Abwischen oder Abtrocknen ein weiches Tuch. Verwenden Sie bei den Plastikröhrchen keine Papierhandtücher oder Tissue-Papier, da dieses sie zerkratzen kann. Spülen Sie mit sauberem Wasser (vorzugsweise entsalztes Wasser).
- Wenn Sie titrieren, zählen Sie bitte jeden Tropfen verwendete Titersubstanz. Halten Sie die Tropfpipette senkrecht. Schwenken Sie den Lösungsbehälter nach der Hinzufügung jedes Tropfens.
- Das Ergebnis kann in Grains je Gallone (gpg) ausgedrückt werden, indem man das Ergebnis in mg/L durch 17,1 dividiert.
- Öffnen der PermaChem®-Pulverkissen:
  1. Klopfen Sie mit dem Boden des Kissens auf eine harte Oberfläche.
  2. Öffnen Sie das Kissen und bilden Sie durch Drücken der Seitenkanten einen Ausgießer.
  3. Schutten Sie den Inhalt in die Probe.
- Hach empfiehlt dringend, für optimale Testergebnisse die Genauigkeit des Reagenzes bei jeder neuen Charge von Reagenzien zu überprüfen. Verwenden Sie dazu die diesem Kit beiliegende Standardlösung oder die im Abschnitt *ZUSÄTZLICHE REAGENZIEN UND ZUBEHÖR* aufgeführte Standardlösung. Befolgen Sie die Anweisungen, die jeder Standardlösung beiliegen.

Consejos para la medición e información general sobre el análisis

- Lavar todo el material de laboratorio entre los análisis. La contaminación puede alterar los resultados. Limpiar con detergentes no abrasivos o con un disolvente como el alcohol isopropílico. Utilizar un paño suave para limpiar o secar. No utilizar ni toallitas ni pañuelos de papel para limpiar los tubos de plastico para no rayarlos. Aclarar con agua limpia (preferentemente agua desionizada).
- Al valorar, cuente cada gota de solución valoradora añadida. Mantenga el cuentagotas en posición vertical. Agite el matraz tras añadir cada gota.
- El resultado puede expresarse en granos por galón (gpg) dividiendo el resultado en mg/L entre 17,1 (1 gpg = 17,1 mg/L).
- Para abrir las Cápsulas de Reactivo PermaChem® proceda del siguiente modo:
  1. Golpee ligeramente la parte inferior de la cápsula contra una superficie dura.
  2. Tire de la línea de puntos para abrir.
  3. Abra la cápsula y presione sobre los laterales de la misma hasta que brote el contenido.
  4. Vierta el contenido en la muestra.

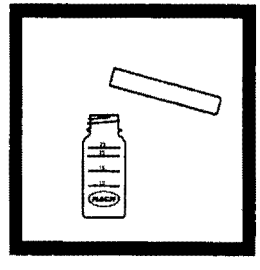
- Para obtener mejores resultados, Hach recomienda encarecidamente comprobar la validez del reactivo con cada nuevo lote. Utilice para ello la solución patrón incluida en este kit o mencionada en la sección de *REACTIVOS Y EQUIPAMIENTO OPCIONALES*. Siga las instrucciones que se incluyen en cada solución patrón.

• High Range Test (20–400 mg/L)
• Technique gamme haute
• Test für den hohen Bereich
• Determinación de alcalinidad, valores altos



1. Fill plastic tube full (to the top) with sample water.

- Remplir le petit tube plastique à ras bord avec l'eau à analyser.
- Füllen Sie das Plastikröhrchen mit Probenwasser voll (bis oben hin).
- Llene hasta el máximo la probeta de plástico con la muestra de agua.

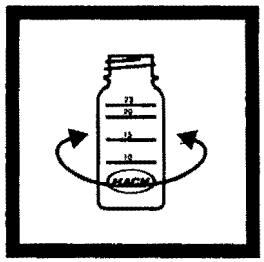


2. Pour the contents of the tube into the square mixing bottle.

- Verser le contenu du tube dans le flacon carré.
- Gießen Sie den Inhalt des Röhrchens in die viereckige Mischflasche.
- Vierta el contenido de la probeta en el matraz.

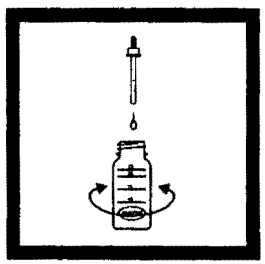
3. Add the contents of one Phenolphthalein Indicator Powder Pillow to the mixing bottle.

- Ajouter le contenu d'un sachet d'indicateur phénolphthaléine au flacon carré.
- Geben Sie den Inhalt eines Phenolphthalein-Pulverkissens in die Mischflasche
- Añada el contenido de una cápsula de indicador fenoftaleina al matraz.



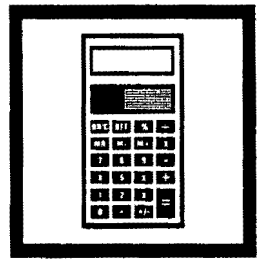
4. Swirl to mix. If the water remains colorless, the phenolphthalein alkalinity is zero. In this case, proceed to Step 7.

- Agiter pour mélanger. Si l'eau reste incolore, l'alcalinité à la phénolphthaléine est zéro. Dans ce cas, passer à l'étape 7.
- Schwenken Sie sie zum Vermischen. Bleibt das Wasser farblos, so ist die Phenolphthalein-Alkalität (p-Wert) Null. In diesem Falle fahren Sie mit Schritt 7 fort.
- Agite para mezclar. Si la muestra permanece incolora la alcalinidad en presencia de fenoftaleina es cero. En este caso continúe en el punto 7.



5. If the sample turns pink, add Sulfuric Acid Standard Solution one drop at a time. Count each drop. Swirl the mixing bottle after each drop is added. Add drops until the sample turns colorless.

- Si l'échantillon devient rose, ajouter goutte à goutte la solution d'acide sulfurique en comptant les gouttes. Agiter le flacon après chaque goutte. Continuer jusqu'à virage de l'indicateur du rose à l'incolore.
- Wird die Probe rosa, geben Sie Schwefelsäure-Standardlösung hinzu, und zwar jeweils einen Tropfen. Zählen Sie jeden Tropfen. Schwenken Sie die Mischflasche nach jedem Tropfen. Geben Sie Tropfen hinzu, bis die Probe farblos wird.
- Si la muestra se vuelve rosa, añada gota a gota la solución patrón de acido sulfúrico. Cuente cada gota añadida. Agite el matraz tras añadir cada gota. Añada gotas hasta que la muestra se vuelva incolora.



6. Multiply by 20 the number of drops of titrant used. This is the mg/L of phenolphthalein alkalinity as calcium carbonate ($CaCO_3$).

mg/L $CaCO_3$ phenolphthalein alkalinity = number of drops x 20

- L'alcalinité à la phénolphthaléine en mg/L de carbonate de calcium ($CaCO_3$) est obtenue en multipliant le nombre de gouttes par 20.

mg/L d'alcalinité à la phénolphthaléine = nombre de gouttes x 20

- Multiplizieren Sie die Anzahl der Tropfen verbrauchte Titersubstanz mit 20. Das sind die mg/L des p-Wertes, als Calciumcarbonat ($CaCO_3$) ausgedrückt.

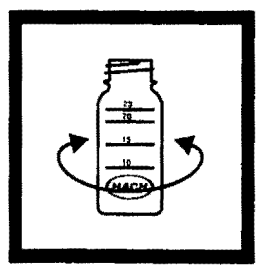
mg/L $CaCO_3$ Phenolphtalein-Alkalität = Anzahl der Tropfen x 20

- Multiplique el número de gotas de solución valoradora (ácido sulfúrico) utilizadas por 20 para obtener la alcalinidad del agua en presencia de fenoftaleina expresada en mg/L de carbonato cálcico ($CaCO_3$).

mg/L de alcalinidad en presencia de fenoftaleina = número de gotas x 20

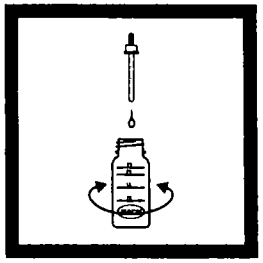
7. Add the contents of one Bromcresol Green-Methyl Red Indicator Powder Pillow to the mixing bottle.

- Ajouter le contenu d'un sachet d'indicateur vert de bromocrésol-rouge de méthyle au flacon carré.
- Geben Sie den Inhalt eines Bromcresolgrün-Methylrot-Indikator-Pulverkissens in die Mischflasche.
- Añada el contenido de una cápsula de indicador verde de bromocresol-rojo de metilo al matraz para mezclar.



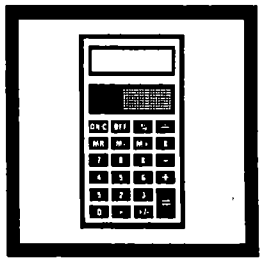
8. Swirl to mix.

- Agiter pour mélanger.
- Schwenken Sie zum Vermischen.
- Agite para mezclar.



9. Add Sulfuric Acid Standard Solution one drop at a time. Count each drop. Swirl the mixing bottle after each drop is added. Add drops until the sample turns pink.

- Ajouter goutte à goutte la solution d'acide sulfurique en comptant les gouttes. Agiter le flacon après chaque goutte. Continuer jusqu'à virage au rose de l'indicateur.
- Geben Sie Schwefelsäure-Standardlösung dazu, jeweils einen Tropfen. Zählen Sie jeden Tropfen. Schwenken Sie die Mischflasche nach jedem Tropfen. Geben Sie Tropfen hinzu, bis die Farbe der Probe in rosa umschlägt.
- Añada gota a gota una solución patrón de ácido sulfúrico. Cuente cada gota añadida. Agite el matraz tras añadir cada gota. Continúe añadiendo gotas hasta que la muestra se vuelva rosa.



10. Multiply by 20 the total number of drops of titrant used in both steps 5 and 9. This is the total mg/L of methyl orange alkalinity as calcium carbonate ($CaCO_3$).

mg/L $CaCO_3$ methyl orange alkalinity = number of drops x 20

- L'alcalinité totale ou alcalinité à l'orange de méthyle en mg/L de carbonate de calcium ($CaCO_3$) est obtenue en multipliant par 20 le nombre total de gouttes utilisées aux étapes 5 et 9.

mg/L d'alcalinité à l'orange de méthyle = nombre de gouttes x 20

- Multiplizieren Sie die Gesamtzahl der Tropfen der in Schritt 5 und 9 verbrauchten Titersubstanz mit 20. Das sind die gesamten mg/L des m-Wertes, als $CaCO_3$ ausgedrückt.

mg/L $CaCO_3$ m-Wert = Tropfenzahl x 20

- Multiplique por 20 el número total de gotas de solución valoradora utilizadas en el punto 5 y en el 9. Esto resultará en la alcalinidad total del agua en presencia de naranja de metilo expresada en mg/L de $CaCO_3$.

mg/L alcalinidad en presencia de naranja de metilo = número de gotas x 20

• LowRange Test (5–100 mg/L)
• Technique gamme basse
• Test für den niedrigen Bereich
• Determinación de alcalinidad, valores bajos

1. Fill the mixing bottle to the 23-mL mark with the sample water.

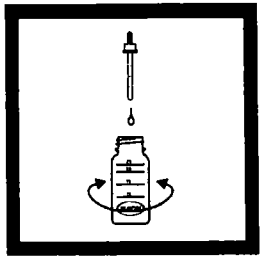
- Remplir le flacon carré jusqu'au trait 23 mL avec l'échantillon d'eau.
- Füllen Sie die Mischflasche bis zur 23 mL-Markierung mit dem Probenwasser.
- Llene la probeta de plástico hasta la marca de 23 mL con la muestra de agua.

2. Add the contents of one Phenolphthalein Indicator Powder Pillow.

- Ajouter le contenu d'un sachet d'indicateur phénolphthaléine au flacon carré.
- Geben Sie den Inhalt eines Phenolphthalein-Pulverkissens hinzu.
- Añada el contenido de una cápsula de indicador fenoftaleina.

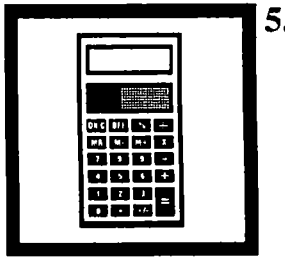
3. Swirl to mix. If the sample remains colorless, the phenolphthalein alkalinity is zero. In this case, proceed to Step 6.

- Agiter pour mélanger. Si l'eau reste incolore, l'alcalinité à la phénolphthaléine est zéro. Dans ce cas, passer à l'étape 6.
- Schwenken Sie zum Vermischen. Bleibt die Probe farblos, so ist der p-Wert Null. In diesem Falle fahren Sie mit Schritt 6 fort.
- Agite hasta mezclar. Si el agua permanece incolora, la alcalinidad en presencia de fenoftaleina es cero. En este caso continúe con el punto 6.



4. If the sample turns pink, add Sulfuric Acid Standard Solution one drop at a time. Count each drop. Swirl the mixing bottle after each drop is added. Add drops until the sample turns colorless.

- Si l'échantillon devient rose, ajouter goutte à goutte la solution d'acide sulfurique en comptant les gouttes. Agiter le flacon après chaque goutte. Continuer jusqu'à virage de l'indicateur du rose à l'incolore.
- Wird die Probe rosa, geben Sie Schwefelsäure-Standardlösung hinzu, und zwar jeweils einen Tropfen. Zählen Sie jeden Tropfen. Schwenken Sie die Mischflasche nach jedem Tropfen. Geben Sie Tropfen hinzu, bis die Probe farblos wird.
- Si la muestra se vuelve rosa, añada gota a gota solución patrón de ácido sulfúrico. Cuente cada gota añadida. Agite el matraz tras añadir cada gota. Continúe añadiendo gotas hasta que la muestra se vuelva incolora.



5. Multiply by 5 the number of drops of titrant used. This is the mg/L of phenolphthalein alkalinity as calcium carbonate ($CaCO_3$).

mg/L $CaCO_3$ phenolphthalein alkalinity = number of drops x 5

- L'alcalinité à la phénolphthaléine en mg/L de carbonate de calcium ($CaCO_3$) est obtenue en multipliant le nombre de gouttes par 5.

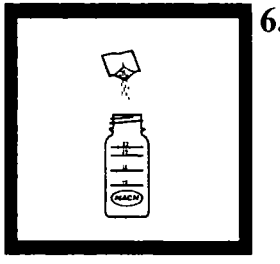
mg/L d'alcalinité à la phénolphthaléine = nombre de gouttes x 5

- Multiplizieren Sie die Zahl der verbrauchten Tropfen Titersubstanz mit 5. Das sind die mg/L des p-Wertes, ausgedrückt als Calciumcarbonat ($CaCO_3$).

mg/L $CaCO_3$ Phenolphtalein-Alkalität = Tropfenzahl x 5

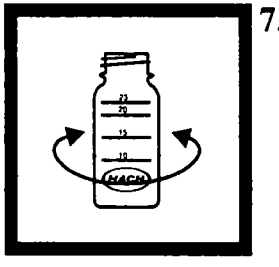
- Multiplique el número de gotas de solución valoradora utilizadas por 5. Para obtener alcalinidad del agua en presencia de fenoftaleina expresada en mg/L de carbonato cálcico.

mg/L alcalinidad en presencia de fenoftaleina = número de gotas x 5



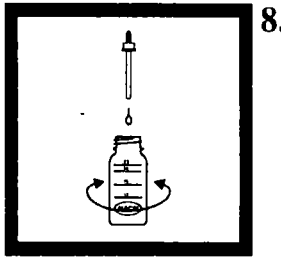
6. Add the contents of one Bromcresol Green-Methyl Red Indicator Powder Pillow to the mixing bottle.

- Ajouter le contenu d'un sachet d'indicateur vert de bromocrésol-rouge de méthyle au flacon carré.
- Geben Sie den Inhalt eines Bromcresolgrün-Methylrot-Indikator-Pulverkissens in die Mischflasche.
- Añada el contenido de una cápsula de indicador verde de bromocresol-rojo de metilo al matraz.



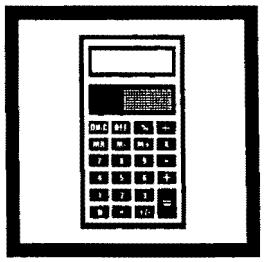
7. Swirl to mix.

- Agiter pour mélanger.
- Schwenken Sie zum Vermischen.
- Agite para mezclar.



8. Add Sulfuric Acid Standard Solution one drop at a time. Count each drop. Swirl the mixing bottle after each drop is added. Add drops until the sample turns pink.

- Ajouter goutte à goutte la solution d'acide sulfurique en comptant les gouttes. Agiter le flacon après chaque goutte. Continuer jusqu'à virage au rose de l'indicateur.
- Geben Sie Schwefelsäure-Standardlösung dazu, jeweils einen Tropfen. Zählen Sie jeden Tropfen. Schwenken Sie die Mischflasche nach jedem Tropfen. Geben Sie Tropfen hinzu, bis die Farbe der Probe in rosa umschlägt.
- Añada gota a gota la solución patrón de acido sulfúrico. Cuente cada gota añadida. Agite el matraz tras añadir cada gota. Continúe añadiendo gotas hasta que la muestra se vuelva rosa.



9. Multiply by 5 the total number of drops of titrant used in both steps 4 and 8. This is the total mg/L of methyl orange alkalinity as calcium carbonate ($CaCO_3$).

mg/L $CaCO_3$ methyl orange alkalinity = number of drops x 5

• L'alcalinité totale ou alcalinité à l'orange de méthyle en mg/L de carbonate de calcium ($CaCO_3$) est obtenue en multipliant par 5 le nombre total de gouttes utilisées aux étapes 4 et 8.

mg/L d'alcalinité à l'orange de méthyle = nombre de gouttes x 5

• Multiplizieren Sie die Zahl der in Schritt 4 und 8 verbrauchten Tropfen Titersubstanz mit 5. Das sind die gesamten mg/L des m-Wertes, als $CaCO_3$ ausgedrückt.

mg/L $CaCO_3$ m-Wert = Tropfenzahl x 5

• Multiplique el número total de gotas de solución valoradora utilizadas en los puntos 4 y 8 por 5. Esto resultará en alcalinidad total de la muestra en presencia de naranja de metilo expresada en mg/L de carbonato cálcico ($CaCO_3$).

mg/L alcalinidad en presencia de naranja de metilo = número de gotas x 5

REPLACEMENTS

Description	Unit	Cat. No.
Alkalinity Reagent Set, Drop Count Titration 0–400 mg/L as $CaCO_3$ *Includes*: (1) 942-99, (1) 943-99, (1) 23497-32	100 tests	24374-01
Bottle, mixing, glass	6/pkg	2327-06
Bromcresol Green-Methyl Red Powder Pillows	100/pkg	943-99
Instruction Card, AL-AP Test Kit	each	24443-89
Measuring Tube, plastic, 5.83 mL	each	438-00
Phenolphthalein Powder Pillows	100/pkg	942-99
Sulfuric Acid Standard Solution, 0.035 N	100 mL MDB[1]	23497-32

REACTIFS ET PIECES DE RECHANGE

Désignation	Unité	Réf. №
Kit de réactifs alcalinité, titrage au compte-gouttes 0–400 mg/L en $CaCO_3$ *Comprenant*: (1) 942-99, (1) 943-99, (1) 23497-32	100 tests	24374-01
Flacon carré en verre	6/paq	2327-06
Indicateur vert de bromocrésol-rouge de méthyle en sachets	100/paq	943-99
Mode d'emploi de la trousse AL-AP	1	24443-89
Tube de mesure en plastique, 5,83 mL	1	438-00
Indicateur phénolphthaléine en sachets	100/paq	942-99
Acide sulfurique, solution 0,035 N	100 mL CGG[2]	23497-32

1 Marked Dropping Bottle
2 Compte-gouttes gradué

VERBRAUCHSMATERIAL UND ERSATZTEILE

Beschreibung	Einheit	Kat. Nr.
Alkalitätsreagenzsatz, Tropfenzähl-Titration 0–400 mg/L als $CaCO_3$ *Enthält*: (1) 942-99, (1) 943-99, (1) 23497-32	100 Tests	24374-01
Mischflasche	6/Stck	2327-06
Bromkresolgrün-Methylrot-Pulverkissen	100/Stck	943-99
Anleitungskarte, AL-AP Test Kit	1	23334-89
Meßröhrchen, Plastik, 5,83 mL	1	438-00
Phenolphthalein-Pulverkissen	100/Stck	942-99
Schwefelsäure-Standardlösung, 0,035 N	100 mL MT[3]	23497-32

REACTIVOS Y MATERIALES

Descripción	Unidad	Nº Ref.
Kit de reactivos para la determinación de alcalinidad por valoración, en mg/L de $CaCO_3$ (0–400 mg/L) *Incluye:* (1) 942-99, (1) 943-99, (1) 23497-32	Para 100 análisis	24374-01
Matraz para mezclar, cuadrado	6/lote	2327-06
Cápsulas de indicador verde bromocresol-rojo de metilo	100/lote	943-99
Tarjeta de Instrucciones, Juego de Prueba AL-AP	1	24443-89
Probeta de plástico, 5,83 mL	1	438-00
Cápsulas de indicador fenoftaleina	100/lote	942-99
Solución patrón de ácido sulfúrico	100 mL FCG[4]	23497-32

3 Markierte Tropfflasche
4 Frasco cuentagotas graduado

OPTIONAL REAGENTS AND EQUIPMENT

Description	Unit	Cat. No.
Alkalinity Standard Solution, 0.500 N, 2-mL PourRite™ Ampule	20/pkg	14278-20

REACTIFS ET EQUIPEMENTS OPTIONNELS

Désignation	Unité	Réf. Nº
Solution étalon alcalinité, 0.500 N, ampoule PourRite™ 2 mL	20/paq	14278-20

ZUSÄTZLICHE REAGENZIEN UND ZUBEHÖR

Beschreibung	Einheit	Kat. Nr.
Alkalitäts-Standardlösung, 0,500 N, 2 mL-PourRite™-Ampulle	20/Stck	14278-20

REACTIVOS Y EQUIPAMIENTO OPCIONALES

Descripción	Unidad	Nº Ref.
Solución patrón de Alcalinidad, 0,500N, Ampolla PourRite™ de 2mL	20/lote	14278-20

- **Pour assistance technique, informations de prix ou informations pour commander, contactez HACH Company ou votre distributeur HACH.**
- **Technische Unterstützung, aktuelle Preisauskünfte und Bestellhilfe erhalten Sie bei Ihrer HACH Vertretung.**
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Outside the U.S.A. - **Contact the HACH office or distributor serving you.**

te/dk 4/97 1ed

HYDROGEN SULFIDE TEST KIT (INCLUDING PRETREATMENT)
Range 0-0.55, 0-2.25 and 0-11.25 mg/L
FOR WATER AND WASTEWATER ANALYSIS
MODEL HS - WR CAT. NO. 2238-01

HACH

TO ENSURE ACCURATE RESULTS READ CAREFULLY BEFORE PROCEEDING.

This procedure determines the total sulfide present in the samples as hydrogen sulfide or acid-soluble metallic sulfides. Soluble sulfide can be determined by centrifuging some of the original sample in completely filled and tightly capped tubes (no trapped air) and performing the test on the clear supernatant. The difference between the soluble sulfide result and the total sulfide obtained on an uncentrifuged sample equals the insoluble sulfide. The results of centrifuging can be approximated in the field by briefly allowing the particulate matter to settle.

A test for total sulfide usually is performed only on sample such as potable or well water which are free of suspended solids. The Pretreatment Instructions provide a means of determining total sulfide in a colored and/or turbid sample. Substitute the pretreated sample for the demineralized water in Step 1 of the instructions.

WARNING:The chemicals in this kit may be hazardous to the health and safety of the user if inappropriately handled. Please read all warnings before performing the test and use appropriate safety equipment.

HACH COMPANY, P.O. BOX 389, LOVELAND, COLORADO 80359
TELEPHONE: WITHIN U.S. 800-227-4224, OUTSIDE U.S. 970-669-3050, TELEX: 160840

Strong reducing agents may diminish the intensity of the blue color development completely. See Section 427B of APHA Standard Methods, 15th edition, for details.

Extremely high concentrations of sulfide also will inhibit the color formation and require a sample dilution.

The test for sulfides should be performed as promptly as possible after the water sample has been taken. Consult APHA Standard Methods if sample preservation is necessary.

PRETREATMENT INSTRUCTIONS

1. Fill the glass measuring bottle about half full with water to be tested. Shake vigorously. Empty the bottle and repeat procedure.

2. Fill the glass measuring bottle to the 25 mL mark with the water sample.

3. Add Bromine Water (approximately three drops) until a faint but permanent yellow-brown color appears. Swirl to mix. Allow to stand for three minutes. Rinse the dropper several times with clear water before storing.

4. Add one drop of Phenol Solution to the sample. Swirl to mix. Rinse the dropper several times in clear water before storing.

5. Use this treated solution in the appropriate test range.

LOW RANGE 0 - .55 mg/L

1. Rinse one of the square mixing bottles with demineralized water or the pretreated water sample. Fill to the 25 mL mark. This is the blank solution.
2. Rinse the second square mixing bottle with the sample to be tested. Fill to the 25 mL mark. Avoid aeration of the sample during the test procedure.
3. Fill the dropper to the 1 mL mark with Sulfide 1 Reagent. Add 1 mL of Sulfide 1 Reagent to each mixing bottle. Swirl to mix as shown in Figure 1.
4. Fill the second dropper to the 1 mL mark with Sulfide 2 Reagent. Add 1 mL of Sulfide 2 Reagent to each mixing bottle. Swirl to mix. The prepared sample immediately will turn pink and then blue if sulfide is present. Allow the sample to stand for five minutes for full color development.
5. Insert the lengthwise adapter into the comparator as shown in Figure 2.
6. Fill one sample tube exactly to the line below the number 46600 with the prepared sample.
7. Fill the second sample tube with the prepared blank to the line below the number 46600.
8. Place the prepared sample from Step 6 into the right top opening of the comparator. (Prepared Sample Position in Figure 2)
9. Place the prepared blank from step 7 into the left top opening of the comparator. (Prepared Blank in Figure 2)

10. Hold the comparator with the tubes tops pointing to a window or light source as shown in Figure 2a. View through the openings in the front of the comparator. When viewing use care to not spill samples from unstoppered tubes.
11. Rotate the disc to obtain a color match. Read the total sulfide as mg/L S^{2-} from the lower scale through the scale window. To obtain results as mg/L H_2S, multiply the mg/L S^{2-} by 1.06.

MEDIUM RANGE 0 - 2.25 MG/L

1. Rinse one of the square mixing bottles with demineralized water or the pretreated water sample. Fill to the 25 mL mark. This is the blank solution.
2. Rinse the second square mixing bottle with the sample to be tested. Fill to the 25 mL mark. Avoid aeration of the sample during the test procedure.
3. Fill the dropper to the 1 mL mark with Sulfide 1 Reagent. Add 1 mL of Sulfide 1 Reagent to each mixing bottle. Swirl to mix as shown in Figure 1.
4. Fill the second dropper to the 1 mL mark with Sulfide 2 Reagent. Add 1 mL of Sulfide 2 Reagent to each mixing bottle. Swirl to mix. The prepared sample immediately will turn pink and then blue if sulfide is present. Allow the sample to stand for five minutes for full color development.
5. If the lenthwise adapter is in place, remove it from the comparator box.
6. Fill one sample tube to the frosted area with the prepared sample.

7. Fill the second sample tube with the prepared blank to the frosted area.
8. Place the prepared sample from Step 6 into the right top opening of the comparator. (Prepared Sample Position in Figure 2)
9. Place the prepared blank from Step 7 into the left top opening of the comparator. (Prepared Blank in Figure 2)
10. Hold the comparator up to a light source such as a window, the sky, or a lamp and view through the openings in the front of the comparator.
11. Rotate the disc to obtain a color match. Read the total sulfide as mg/L S^{2-} from the **upper** scale through the scale window. To obtain the results as mg/L H_2S, multiply the mg/L S^{2-} by 1.06.

HIGH RANGE 0 - 11.25 MG/L

1. Rinse one of the square mixing bottles with demineralized water or the pretreated water sample. Fill to the 25 mL mark. This is the blank solution.
2. Rinse the second square mixing bottle with the sample to be tested. Fill to the 25 mL mark. Avoid aeration of the sample during the test procedure.
3. Fill the dropper to the 1 mL mark with Sulfide 1 Reagent. Add 1 mL of Sulfide 1 Reagent to each mixing bottle. Swirl to mix as shown in Figure 1. Repeat Step 3 again by adding a second 1 mL of Sulfide 1 to each bottle.
4. Fill the second dropper to the 1 mL mark with Sulfide 2 Reagent. Add 1 mL of Sulfide 2 Reagent to each mixing bottle. Swirl to mix. Repeat Step 4 again by adding a second 1 mL of Sulfide 2 to each bottle. The prepared sample immediately will turn pink and then blue if sulfide is present.
5. If the lengthwise adapter is in place, remove it from the comparator.
6. Fill one round bottomed tube to the 5 mL mark with the prepared sample.
7. Insert a clean viewing tube insert into the sample tube (from Step 6). Place this prepared sample into the right top opening of the comparator. (Prepared Sample Position in Figure 3)
8. Fill the second round bottomed tube with the prepared blank to the 5 mL mark.
9. Insert a clean viewing tube insert into the blank tube (from Step 8). Place this prepared blank into the left top opening of the comparator. (Prepared Blank Position in Figure 3)
10. Hold the comparator up to a light source such as a window, the sky, or a lamp and view through the openings in front of the comparator. Rotate the disc until a color match is obtained. Read the value from the **upper** scale.
11. Multiply the value by 5.0 to obtain mg/L S^{2-}.
12. To obtain the results as mg/L H_2S, multiply the mg/L S^{2-} by 1.06.

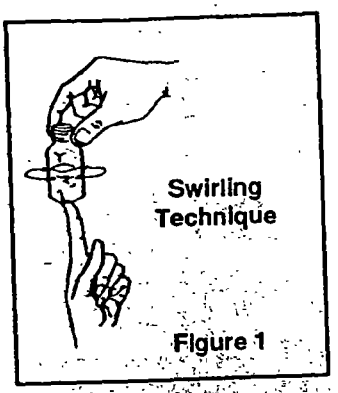


Figure 1

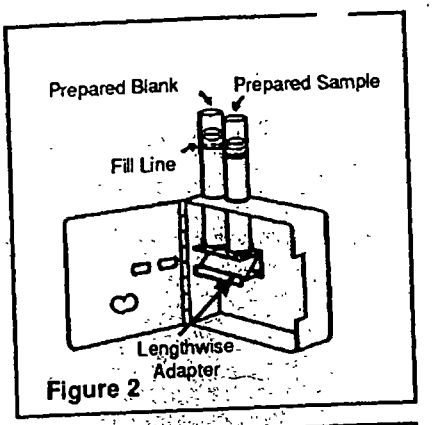


Figure 2

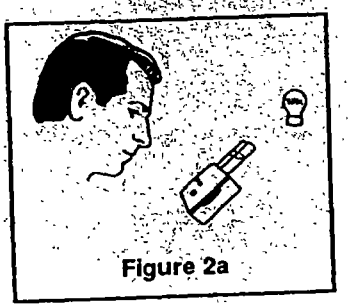


Figure 2a

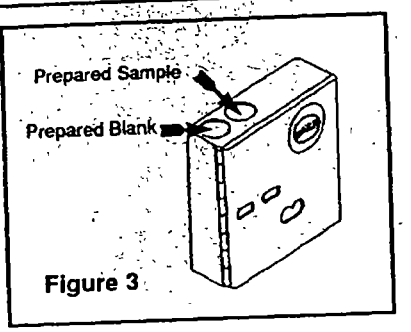


Figure 3

HY

REPLACEMENTS

Cat. No.	Description	Unit
2211-20	Bromine Water	29 mL (oz)
1816-37	Sulfide 1 Reagent	118 mL (4oz)MDB*
1817-37	Sulfide 2 Reagent	118 mL (4oz)MDB*
272-28	Demineralized Water	118 mL (4oz)
2112-20	Phenol Solution	29 mL (oz)
23510-00	Bottle, square mixing	each
1732-00	Color Comparator	each
21945-00	Color Disc, Sulfide	each
46600-04	Viewing tube, plastic	pkg/4
6080-00	Dropper, plastic	each
24122-00	Lenthwise viewing adapter	each
14480-00	Stopper for viewing tube	pkg/6
21288-00	Viewing tube insert, optical	each
21289-00	Tube, viewing 18 x 100 mL with 5mL mark	each

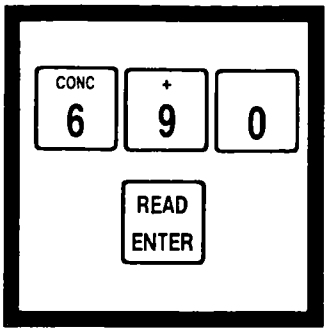
*Marked dropping bottle

12/91 Made in U.S.A.

SULFIDE (0 to 0.600 mg/L S^{2-})

For water, wastewater and seawater

Methylene Blue Method*; EPA Approved



1. Enter the stored program number for sulfide (S^{2-}).

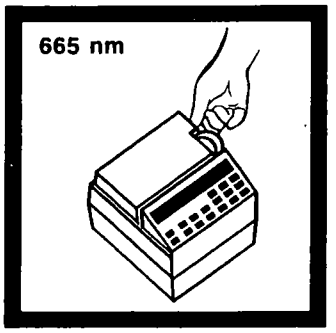
Press: **6 9 0 READ/ENTER**

The display will show:
DIAL nm TO 665

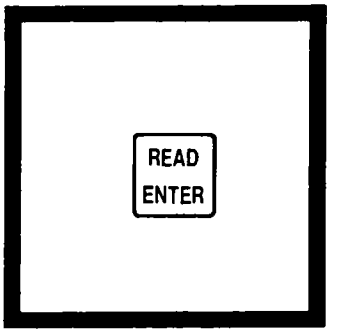
Note: DR/2000s with software versions 3 0 and greater will display "P" and the program number.

Note: Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.

Note: Samples must be analyzed immediately and cannot be preserved for later analysis. Avoid excessive agitation.

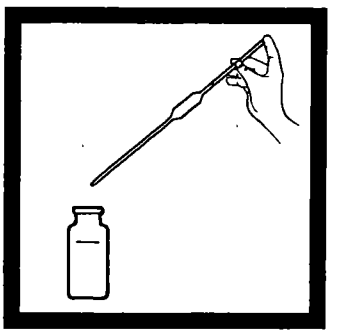


2. Rotate the wavelength dial until the small display shows:
665 nm



3. Press: **READ/ENTER**

The display will show:
mg/l S^{2-}

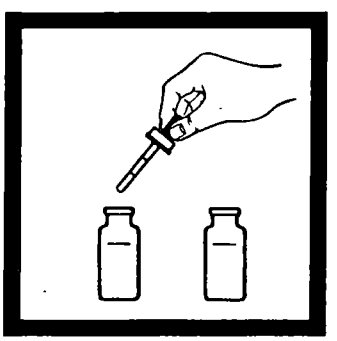


4. Fill a sample cell (the prepared sample) with 25 mL of sample.

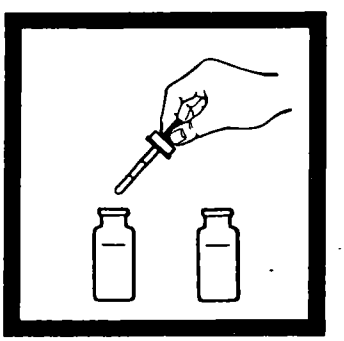
Note: For turbid samples, see Interferences below for pretreatment instructions.

Note: Excessive agitation will cause loss of sulfide Use a pipet to minimize sulfide loss.

5. Fill another sample cell (the blank) with 25 mL of deionized water.

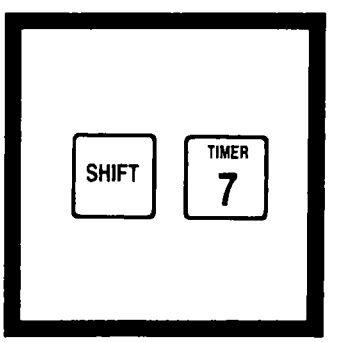


6. Add 1.0 mL of Sulfide 1 Reagent to each cell. Swirl to mix.



7. Add 1.0 mL of Sulfide 2 Reagent to each cell. Immediately swirl to mix.

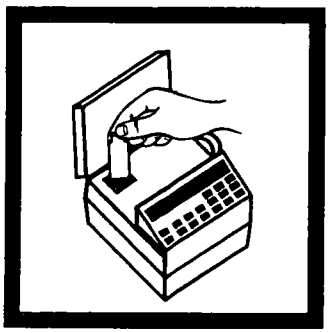
Note: A pink color will develop. The solution will turn blue if sulfide is present.



8. Press: **SHIFT TIMER**

A five-minute reaction time will begin.

*Adapted from *Standard Methods for the Examination of Water and Wastewater*

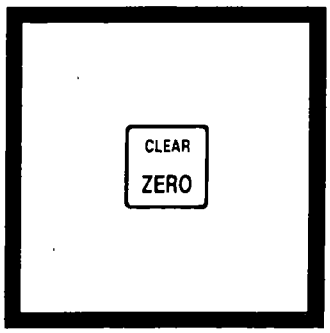


9. When the timer beeps, the display will show:

mg/l S^{2-}

Place the blank into the cell holder. Close the light shield.

Note: The Pour-Thru Cell can be used with this procedure.



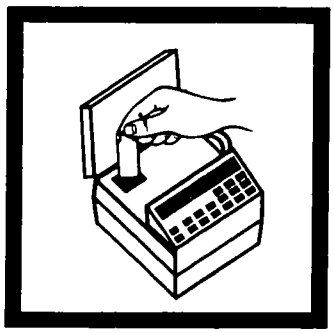
10. Press: **ZERO**

The display will show:

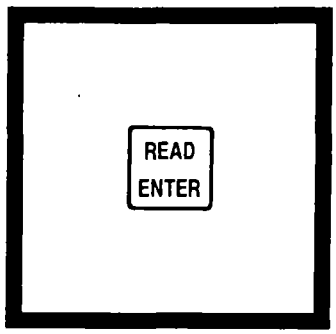
WAIT

then:

0.000 mg/l S^{2-}



11. Immediately place the prepared sample into the cell holder. Close the light shield.



12. Press: **READ/ENTER**

The display will show:

WAIT

then the result in mg/L sulfide (S^{2-}) will be displayed.

Note: In the constant-on mode, pressing READ/ENTER is not required WAIT will not appear When the display stabilizes, read the result.

ACCURACY CHECK

Standard Solution Method

Sulfide standard solutions are very unstable and should be prepared from sodium sulfide and standardized as described in *Standard Methods for the Examination of Water and Wastewater,* 17th ed., page 4-196.

PRECISION

In a single laboratory using a standard solution of 0.250 mg/L sulfide and two lots of reagent with the DR/2000, a single operator obtained a standard deviation of ± 0.003 mg/L sulfide.

INTERFERENCES

For turbid samples, a sulfide-free blank should be prepared as follows. Use it in place of deionized water in Step 5.

a) Measure 25 mL of sample into a flask.

b) Add Bromine Water dropwise until a yellow color remains.

c) Add Phenol Solution dropwise until the yellow color just disappears. Use of this blank solution will compensate for turbidity in the sample.

Strong reducing agents such as sulfite, thiosulfate and hydrosulfite interfere by reducing the blue color or preventing its development. High concentrations of sulfide may inhibit full color development, requiring a dilution of the sample. Some loss of sulfide may occur when the sample is diluted.

SUMMARY OF METHOD

Hydrogen sulfide and acid-soluble metal sulfides react with N,N-dimethyl-p-phenylenediamine oxalate to form methylene blue. The intensity of the blue color is proportional to the sulfide concentration. See *Chemical Procedures Explained*, Publication 7013, for more information.

High sulfide levels in oil-field waters may be determined after proper dilution.

Determine soluble sulfides by centrifuging the sample in completely filled, capped tubes and analyzing the supernatant. Insoluble sulfides are then estimated by subtracting the soluble sulfide concentration from the total sulfide result.

REQUIRED REAGENTS

	Cat. No.
Sulfide Reagent Set (100 Tests)	22445-00
Includes: (2) 1816-14, (2) 1817-14	

Description	Quantity Required Per Test	Unit	Cat. No.
Sulfide 1 Reagent	2 mL	118 mL MDB	1816-37
Sulfide 2 Reagent	2 mL	118 mL MDB	1817-37
Water, deionized	25 mL	3.78 L	272-17

REQUIRED APPARATUS

Cylinder, graduated, 25 mL	1	each	508-40
Pipet, volumetric, 25 mL	1	each	515-40
Pipet Filler, safety bulb	1	each	14651-00

OPTIONAL REAGENTS

Bromine Water, 30 g/L	29 mL	2211-20
Phenol Solution, 30 g/L	29 mL	2112-20
Sodium Sulfide, hydrate	114 g	785-14

OPTIONAL APPARATUS

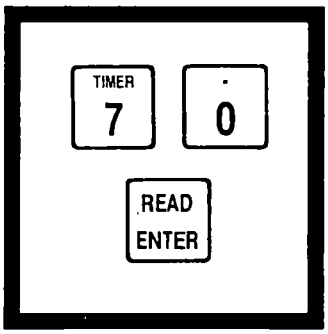
Dropper, for 1 oz bottle	each	2258-00
Flask, erlenmeyer, 50 mL	each	505-41
Pour-Thru Cell Assembly Kit	each	45215-00
Standard Methods for the Examination of Water and Wastewater	each	22708-00

For additional ordering information, see final section.
In the U.S.A. call 800-227-4224 to place an order.

CHLORIDE (0 to 20.0 mg/L Cl^-)

For water and wastewater

Mercuric Thiocyanate Method*



1. Enter the stored program number for Chloride (Cl^-).

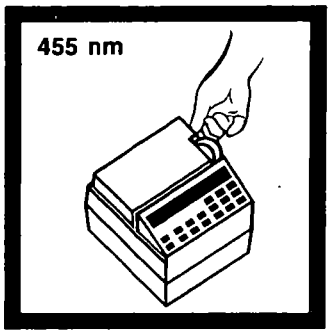
Press: **7 0 READ/ENTER**

The display will show:
DIAL nm TO 455

Note: DR/2000s with software versions 3.0 and greater will display "P" and the program number.

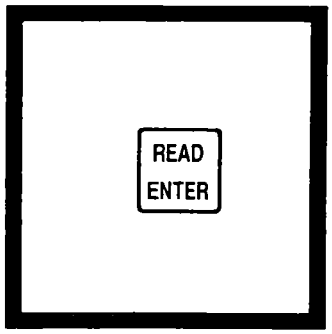
Note: Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.

Note: Samples can be stored for at least 28 days at room temperature in glass or plastic bottles.



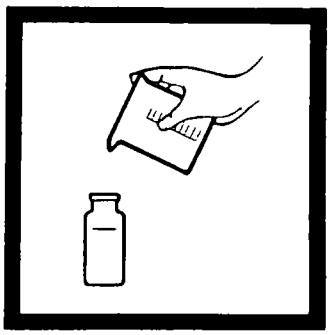
2. Rotate the wavelength dial until the small display shows:
455 nm

Note: Approach the wavelength setting from higher to lower values



3. Press: **READ/ENTER**

The display will show:
mg/l Cl^-

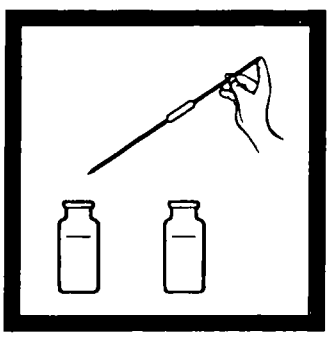


4. Fill a sample cell (the prepared sample) with 25 mL of sample.

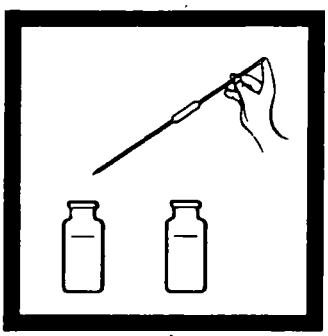
Note: Filter turbid samples through a moderately rapid filter paper before analysis.

Note: For proof of accuracy, use a 10.0 mg/L chloride standard solution (preparation given in the Accuracy Check) in place of the sample.

5. Fill another cell (the blank) with 25 mL of deionized water.

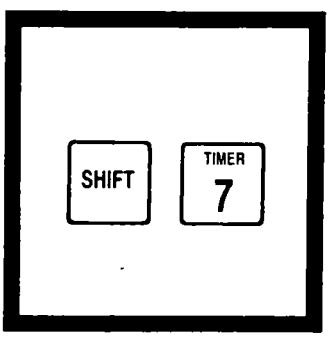


6. Pipet 2.0 mL of Mercuric Thiocyanate Solution into each cell. Swirl to mix.



7. Pipet 1.0 mL of Ferric Ion Solution into each sample cell. Swirl to mix.

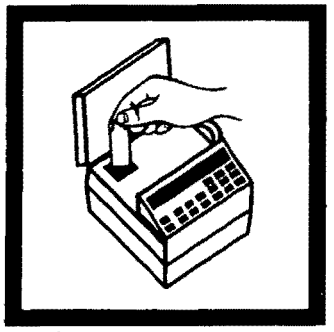
Note: An orange color will develop if chloride is present.



8. Press: **SHIFT TIMER**

A two-minute period will begin.

*Adapted from Zall, et. al., *Analytical Chemistry*, 28 (11) 1665 (1956)

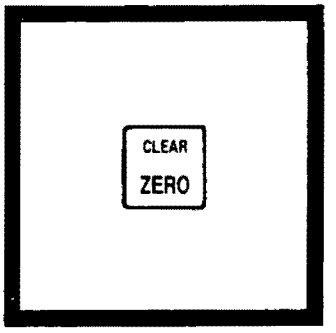


9. When the timer beeps, the display will show:

mg/l Cl^-

Place the blank into the cell holder. Close the light shield.

Note: The Pour-Thru Cell can be used with this procedure.



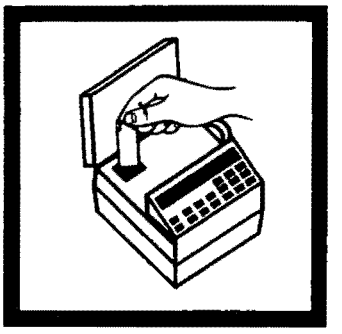
10. Press: **ZERO**

The display will show:

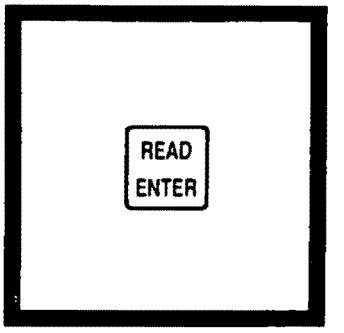
WAIT

then:

0.0 mg/l Cl^-



11. Place the prepared sample into the cell holder. Close the light shield.



12. Press: **READ/ENTER**

The display will show:

WAIT

then the result in mg/L chloride (Cl^-) will be displayed.

Note: In the constant-on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the results.

ACCURACY CHECK

Standard Additions Method

a) Use the TenSette Pipet to add 0.1 mL, 0.2 mL and 0.3 mL of Chloride Standard Solution, 1000 mg/L as Cl^-, to each of three 25-mL water samples. Mix each thoroughly.

b) Analyze each sample as described above.

c) The chloride concentration should increase 4.0 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section 1) for more information.

Standard Solution Method

Prepare a 10.0 mg/L chloride standard solution by diluting 5.00 mL of Chloride Standard Solution, 1000 mg/L to 500 mL with deionized water.

PRECISION

In a single laboratory using a standard solution of 10 mg/L chloride and two lots of reagent with the DR/2000, a single operator obtained a standard deviation of ± 0.3 mg/L chloride.

INTERFERENCES

The pH of the sample after addition of reagents should be about 2. If the sample is strongly acid or alkaline, adjust a portion of sample before testing to a pH of about 7. Use either 5.0 N Sodium Hydroxide Standard Solution or a 1:5 dilution of perchloric acid. Use pH paper, as most pH electrodes will contaminate the sample with chloride.

SUMMARY OF METHOD

Chloride in the sample reacts with mercuric thiocyanate to form mercuric chloride and liberate thiocyanate ion. Thiocyanate ions react with the ferric ions to form an orange ferric thiocyanate complex. The amount of this complex is proportional to the chloride concentration. See *Chemical Procedures Explained*, Publication 7013, for more information. Chloride at these levels also can be determined directly using the Chloride Ion Selective Electrode (Cat. No. 44510-71)

REQUIRED REAGENTS

	Cat. No.
Chloride Reagent Set (50 Tests*)	23198-00
Includes: (1) 22122-14, (1) 22121-31	

Description	Quantity Required Per Test	Unit	Cat. No.
Ferric Ion Solution	2 mL	118 mL	22122-14
Mercuric Thiocyanate Solution	4 mL	236 mL	22121-31
Water, deionized	25 mL	3.78 L	272-17

REQUIRED APPARATUS

Pipet, volumetric, 1.0 mL	1	each	515-35
Pipet, volumetric, 2.0 mL	1	each	515-36
Pipet Filler, safety bulb	1	each	14651-00
OR			
Pipet, TenSette, 0.1 to 1.0 mL	1	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	1	50/pkg	21856-96

OPTIONAL REAGENTS

Chloride Standard Solution, 1000 mg/L as Cl	473 mL	183-11
Perchloric Acid, ACS, 70%	680 g	757-65
Sodium Hydroxide Standard Solution, 5.0 N	59 mL SCDB	2450-26

OPTIONAL APPARATUS

Filter Paper, folded, moderately rapid, 12.5 cm	100/box	692-57
Flask, erlenmeyer, 125 mL	each	505-43
Flask, volumetric, 500 mL	each	547-49
Funnel, filtering, polypropylene, 75 mm	each	1083-68
pH Paper, 1 to 11 pH	5 rolls/pkg	391-33
Pipet, volumetric, Class A, 5 mL	each	14515-37
Pour-Thru Cell Assembly Kit	each	45215-00

Chloride at these levels can be determined directly using the Chloride Ion Selective Electrode (Cat. No. 44510-71)

For additional ordering information, see final section.
In the U.S.A. call 800-227-4224 to place an order.

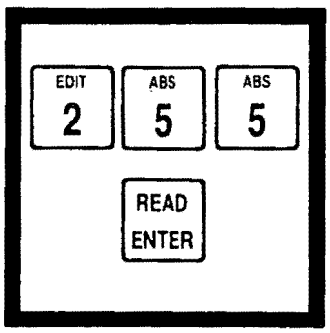
* tests equals 25 samples and 25 blanks

IRON, FERROUS (0 to 3.00 mg/L)

For water, wastewater and seawater

1,10 Phenanthroline Method* (Powder Pillows or AccuVac Ampuls)

USING POWDER PILLOWS



1. Enter the stored program number for ferrous iron, (Fe^{2+})– powder pillows.

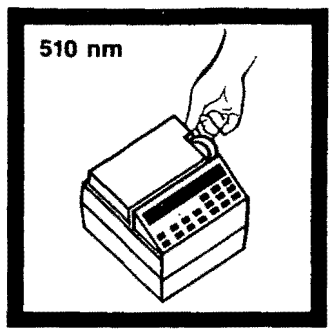
Press: **2 5 5 READ/ENTER**

The display will show:
DIAL nm TO 510

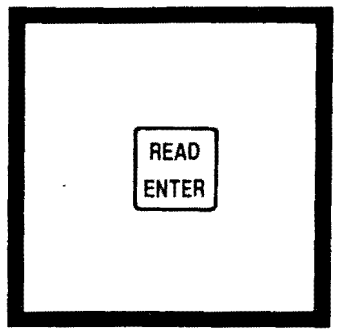
Note: DR/2000s with software versions 3 0 and greater will display "P" and the program number.

Note: Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly The display will show the message in Step 3. Proceed with Step 4.

Note: Analyze samples as soon as possible to prevent air oxidation of ferrous iron to ferric, which is not determined.



2. Rotate the wavelength dial until the small display shows:
510 nm



3. Press: **READ/ENTER**

The display will show:
mg/l Fe^{2+}

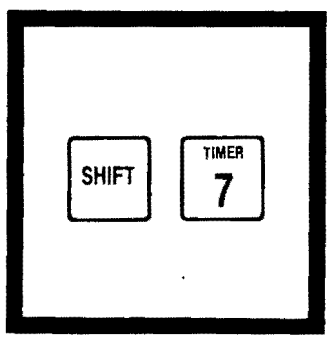
4. Fill a sample cell with 25 mL of sample.

Note: For proof of accuracy, use a 1 0 mg/L ferrous iron standard solution (preparation given in the Accuracy Check) in place of the sample.

5. Add the contents of one Ferrous Iron Reagent Powder Pillow to the sample cell (the prepared sample). Swirl to mix.

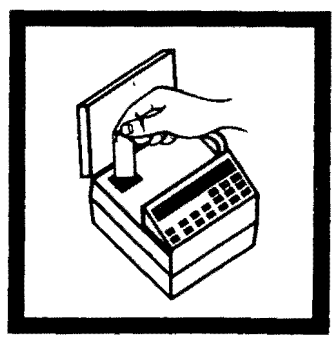
Note: An orange color will form if ferrous iron is present.

Note: Undissolved powder does not affect accuracy.



6. Press: **SHIFT TIMER**

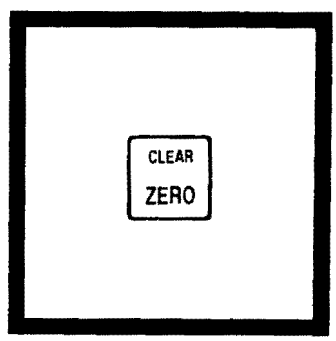
A three-minute reaction period will begin.



7. When the timer beeps, the display will show:
mg/l Fe^{2+}

Fill a second sample cell (the blank) with 25 mL of sample. Place it into the cell holder.

Note: The Pour-Thru Cell can be used with this procedure

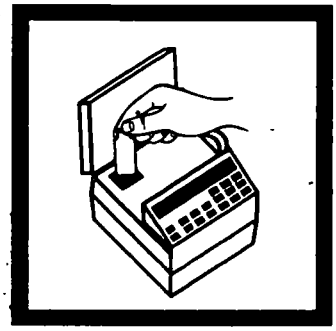


8. Press: **ZERO**

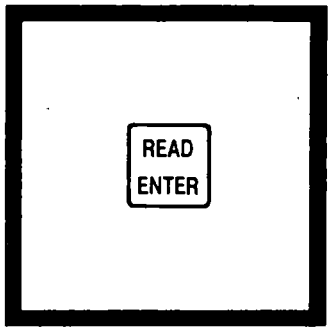
The display will show:
WAIT

then:
0.00 mg/l Fe^{2+}

*Adapted from *Standard Methods for the Examination of Water and Wastewater*



9. Place the prepared sample into the cell holder. Close the light shield.



10. Press: **READ/ENTER**

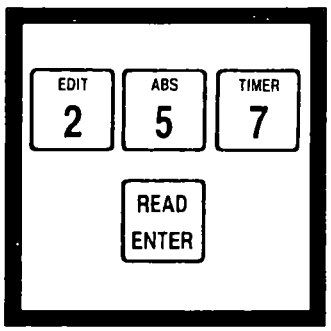
The display will show:

WAIT

then the result in mg/L Fe^{2+} will be displayed.

Note: *In the constant-on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result*

USING ACCUVAC AMPULS



1. Enter the stored program number for ferrous iron (Fe^{2+})–AccuVac ampuls.

Press: **2 5 7 READ/ENTER**

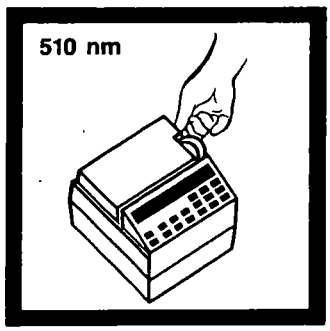
The display will show:

DIAL nm TO 510

Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

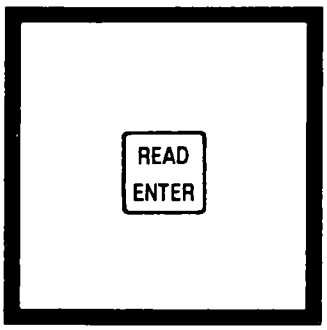
Note: *Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4*

Note: *Analyze samples as soon as possible to prevent air oxidation of ferrous iron to ferric, which is not determined*



2. Rotate the wavelength dial until the small display shows:

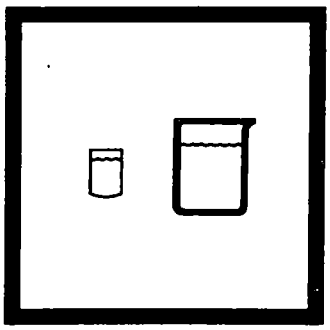
510 nm



3. Press: **READ/ENTER**

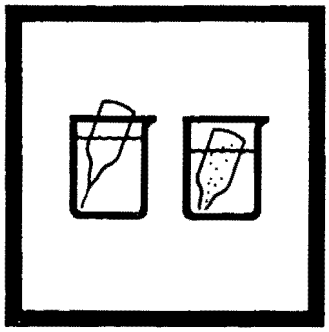
The display will show:

mg/l Fe^{2+} AV



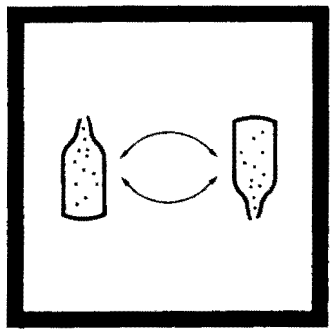
4. Fill a zeroing vial (the blank) with at least 10 mL of sample. Collect at least 40 mL of sample in a 50-mL beaker.

Note: *For proof of accuracy, a 1.0 mg/L ferrous iron standard solution (preparation given in the Accuracy Check) can be used in place of the sample.*



5. Fill a Ferrous Iron AccuVac Ampul with sample.

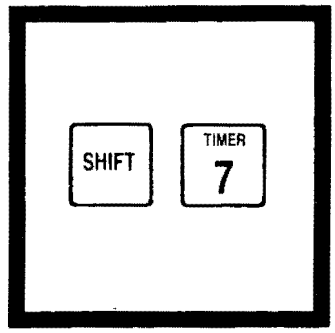
Note: Keep the tip immersed while the ampul fills completely.



6. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

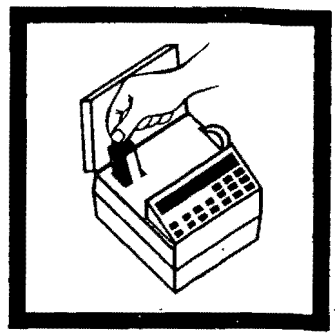
Note: An orange color will form if ferrous iron is present.

Note: Undissolved powder does not affect accuracy.



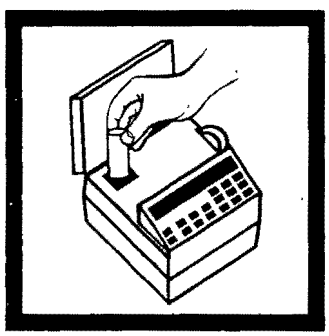
7. Press: **SHIFT TIMER**

A three-minute reaction period will begin.



8. Place the AccuVac Vial Adapter into the cell holder.

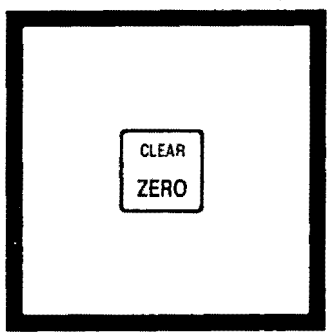
Note: Place the grip tab at the rear of the cell holder.



9. When the timer beeps, the display will show:

mg/l Fe^{2+} AV

Place the blank into the cell holder. Close the light shield.



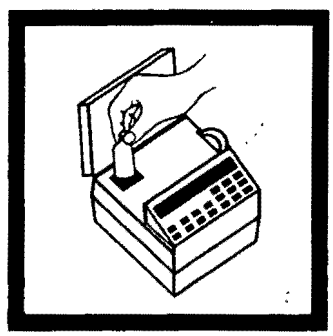
10. Press: **ZERO**

The display will show:

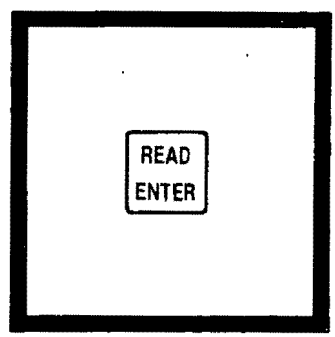
WAIT

then:

0.00 mg/l Fe^{2+} AV



11. Place the AccuVac ampul into the cell holder. Close the light shield.



12. Press: **READ/ENTER**

The display will show:

WAIT

then the result in mg/L Fe^{2+} will be displayed.

Note: In the constant-on mode, pressing READ/ENTER is not required. WAIT will not appear When the display stabilizes, read the result

ACCURACY CHECK

Standard Solution Method

Prepare a ferrous iron stock solution (100 mg/L Fe) by dissolving 0.7022 grams of ferrous ammonium sulfate, hexahydrate, in deionized water. Dilute to 1 liter. Prepare immediately before use. Dilute 1.00 mL of this solution to 100 mL with deionized water to make a 1.0 mg/L standard solution. Prepare this immediately before use.

PRECISION

In a single laboratory using an iron standard solution of 1.000 mg/L Fe^{2+} and two representative lots of reagent with the DR/2000, a single operator obtained a standard deviation of ± 0.006 mg/L Fe^{2+}.

In a single laboratory using a standard solution of 1.000 mg/L Fe^{2+} and two representative lots of AccuVac ampuls with the DR/2000, a single operator obtained a standard deviation of ± 0.009 mg/L Fe^{2+}.

SUMMARY OF METHOD

The 1,10 phenanthroline indicator in Ferrous Iron Reagent reacts with ferrous iron in the sample to form an orange color in proportion to the iron concentration. Ferric iron does not react. The ferric iron (Fe^{3+}) concentration can be determined by subtracting the ferrous iron concentration from the results of a total iron test. See *Chemical Procedures Explained*, Publication 7013, for more information.

REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity Required Per Test	Unit	Cat. No.
Ferrous Iron Reagent Powder Pillows	1 pillow	100/pkg	1037-69

REQUIRED REAGENTS (Using AccuVac Ampuls)

Ferrous Iron Reagent AccuVac Ampuls	1 ampul	25/pkg	25140-25

REQUIRED APPARATUS (Using Powder Pillows)

Clippers, for opening powder pillows	1	each	968-00

REQUIRED APPARATUS (Using AccuVac Ampuls)

Adapter, AccuVac Vial	1	each	43784-00
Beaker, 50 mL	1	each	500-41
Sample Cell, 10-mL, with cap	1	each	21228-00

OPTIONAL REAGENTS

Ferrous Ammonium Sulfate, hexahydrate	113 g	11256-14
Water, deionized	3.78 L	272-17

OPTIONAL APPARATUS

AccuVac Snapper Kit	each	24052-00
Clippers, shears, 7-1/4"	each	23694-00
Flask, volumetric, 100 mL, Class B	each	547-42
Flask, volumetric, 1000 mL, Class B	each	547-53
Pipet, volumetric, 1 mL	each	515-35
Pipet Filler, safety bulb	each	14651-00
Pour-Thru Cell Assembly Kit	each	45215-00

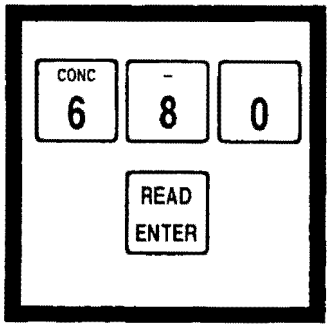
For additional ordering information, see final section.
In the U.S.A. call 800-227-4224 to place an order.

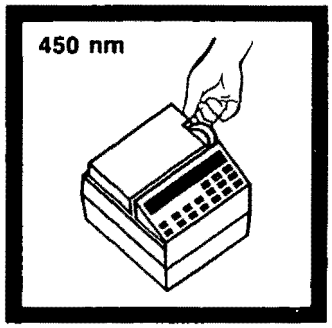
SULFATE (0 to 70 mg/L)

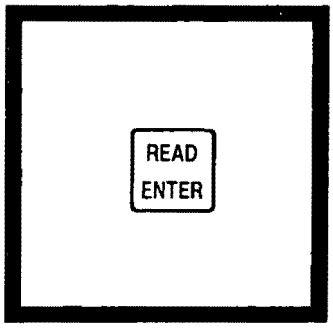
For water, wastewater and seawater

SulfaVer 4 Method* (Powder Pillows or AccuVac Ampuls), EPA Approved

USING POWDER PILLOWS







1. Enter the stored program number for sulfate (SO_4^{2-})–powder pillows.

Press: **6 8 0 READ/ENTER**

The display will show:
DIAL nm TO 450

Note: *DR/2000s with software versions 3.0 and greater will display "P" and the program number.*

Note: *Instruments with software versions 3 0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly The display will show the message in Step 3. Proceed with Step 4.*

Note: *If samples cannot be analyzed immediately, see Sampling and Storage following these steps.*

2. Rotate the wavelength dial until the small display shows:
450 nm

Note: *For best results prepare an instrument calibration for each new lot of SulfaVer 4 Sulfate Reagent Powder Pillows, see Calibration following these steps.*

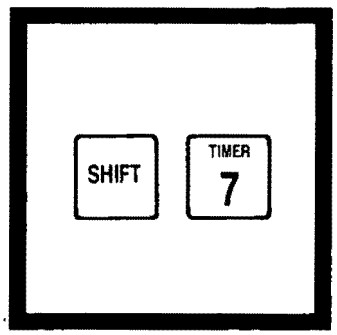
3. Press: **READ/ENTER**

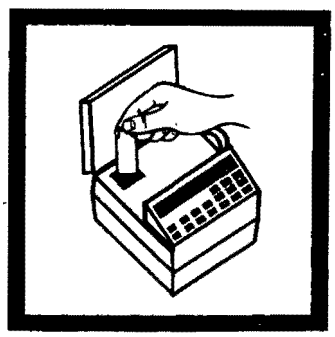
The display will show:
mg/l SO_4^{2-}

4. Fill a sample cell with 25 mL of sample.

Note: *Filter highly colored or turbid samples. Use filtered sample here and in Step 7 Use labware listed under Optional Apparatus.*

Note: *For proof of accuracy, use a 50 mg/L SO_4^{2-} standard solution (preparation given in the Accuracy Check) in place of the sample.*





5. Add the contents of one SulfaVer 4 Sulfate Reagent Powder Pillow to the sample cell (the prepared sample). Swirl to dissolve.

Note: *A white turbidity will develop if sulfate is present.*

Note: *Accuracy is not affected by undissolved powder.*

6. Press: **SHIFT TIMER**

A five minute reaction period will begin.

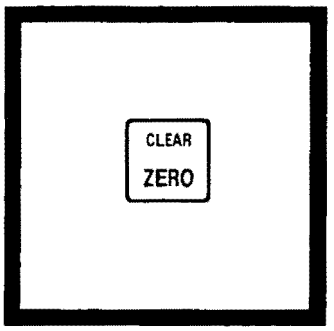
Note: *Allow the cell to stand undisturbed.*

7. When the timer beeps, the display will show:
mg/l SO_4^{2-}
Fill a second sample cell (the blank) with 25 mL of sample.

8. Place the blank into the cell holder. Close the light shield.

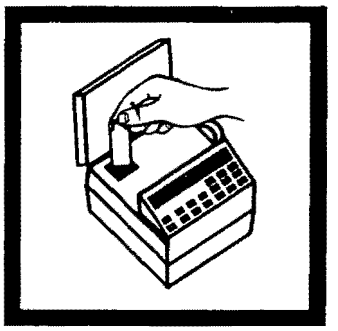
Note: *The Pour-Thru Cell cannot be used with this procedure.*

*Adapted from *Standard Methods for the Examination of Water and Wastewater*

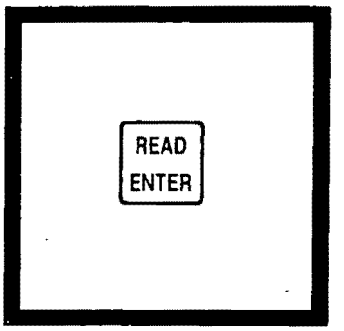


9. Press: **ZERO**

The display will show:
WAIT
then:
0. mg/l SO_4^{2-}



10. Within five minutes after the timer beeps, place the prepared sample into the cell holder. Close the light shield.



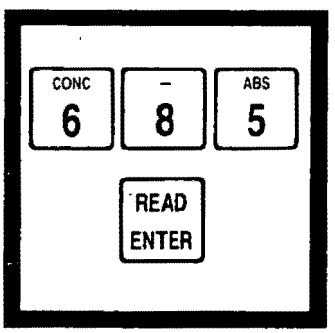
11. Press: **READ/ENTER**

The display will show:
WAIT
then the result in mg/L SO_4^{2-} will be displayed.

Note: Clean the sample cells with soap and a brush.

Note: In the constant-on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.

USING ACCUVAC AMPULS



1. Enter the stored program number for sulfate (SO_4^{2-})–AccuVac Ampuls.

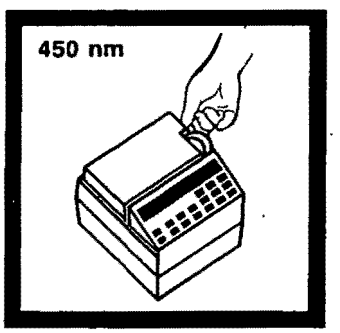
Press: **6 8 5 READ/ENTER**

The display will show:
DIAL nm TO 450

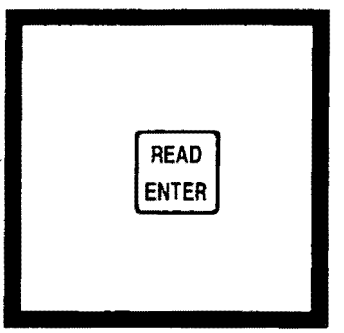
Note: DR/2000s with software versions 3.0 and greater will display "P" and the program number.

Note: Instruments with software versions 3.0 and greater will not display "DIAL nm TO" message if the wavelength is already set correctly. The display will show the message in Step 3. Proceed with Step 4.

Note: If samples cannot be analyzed immediately, see Sampling and Storage below.

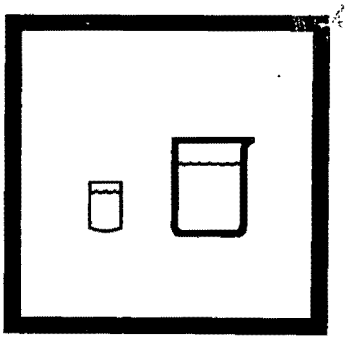


2. Rotate the wavelength dial until the small display shows:
450 nm



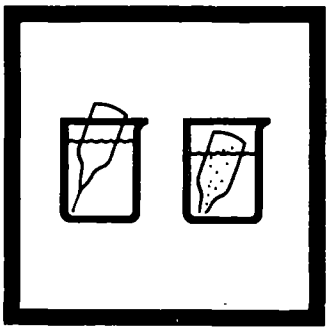
3. Press: **READ/ENTER**

The display will show:
mg/l SO_4^{2-} AV



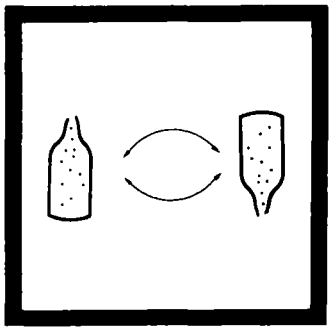
4. Fill a zeroing vial (the blank) with at least 10 mL of sample. Collect at least 40 mL of sample in a 50-mL beaker.

Note: Filter highly colored or turbid samples. Use labware listed under Optional Apparatus.



5. Fill a SulfaVer 4 Sulfate AccuVac Ampul with sample.

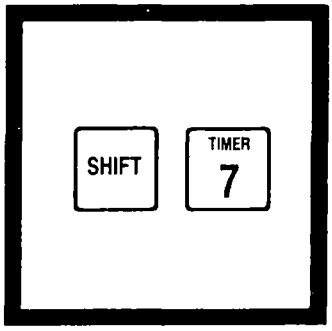
Note: Keep the tip immersed while the ampul fills completely.



6. Quickly invert the ampul several times to mix. Wipe off any liquid or fingerprints.

Note: A white turbidity will form if sulfate is present.

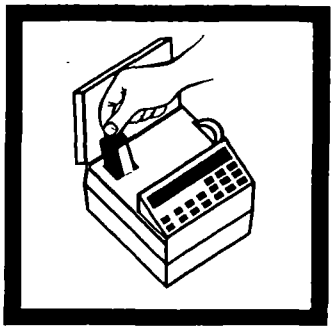
Note: Accuracy is not affected by undissolved powder.



7. Press: **SHIFT TIMER**

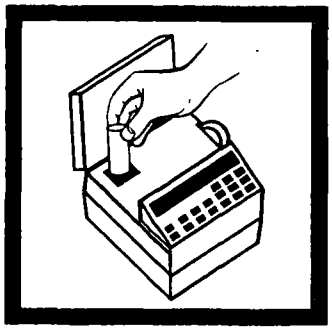
A five-minute reaction time will begin.

Note: Allow the ampul to stand undisturbed.



8. Place the AccuVac Vial Adapter into the cell holder.

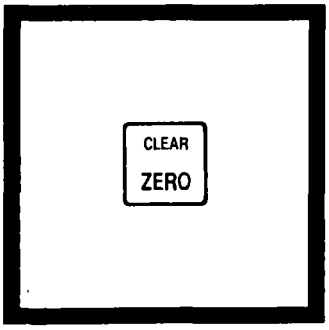
Note: Place the grip tab at the rear of the cell holder



9. When the timer beeps, the display will show:

mg/l SO_4^{2-} AV

Place the blank into the cell holder. Close the light shield.



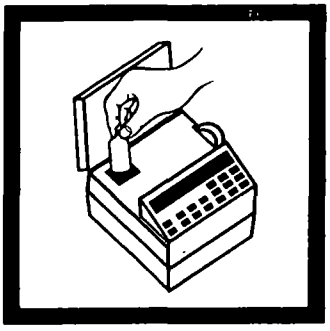
10. Press: **ZERO**

The display will show:

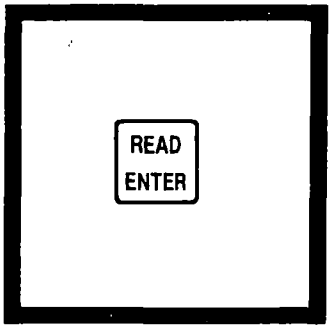
WAIT

then:

0. mg/l SO_4^{2-} AV



11. Within five minutes after the timer beeps, place the AccuVac ampul into the cell holder. Close the light shield.



12. Press: **READ/ENTER**

The display will show:

WAIT

then the result in mg/L SO_4^{2-} will be displayed.

Note: In the constant-on mode, pressing READ/ENTER is not required. WAIT will not appear. When the display stabilizes, read the result.

CALIBRATION

A new calibration may be performed for each lot of SulfaVer 4 Sulfate Reagent Powder Pillows as follows:

a) Prepare standards of 0, 10, 20, 30, 40, 50 and 60 mg/L sulfate by diluting 0, 0.1, 0.2, 0.3, 0.4, 0.5 and 0.6 mL of the contents of a Sulfate Voluette Ampule Standard, 2500 mg/L, to 25.0 mL with deionized water in mixing graduated cylinders. Use a TenSette pipet to measure the standard. Mix well. (Or, pipet 0, 1.0, 2.0, 3.0, 4.0, 5.0 and 6.0 mL of Sulfate Standard Solution into 1000-mL volumetric flasks. Dilute to volume. Mix well. Transfer 25 mL to each test cylinder.)

b) Store the calibration in the instrument memory using the procedure in the Operation section of the instrument manual. Follow the procedure described, choosing a wavelength of 450 nm, the decimal position as 0000, units as mg/L SO_4^{2-}, and a Timer 1 interval of 05:00. Note the program number assigned to the procedure.

c) Add the reagents to the deionized water (0 standard-reagent blank) and to the 10 mg/L standard as described in Steps 4 to 6 above, using the deionized water blank to perform the zero calibration. Enter the sulfate concentration of the first standard (10 mg/L) and measure the absorbance as directed by the instrument. React and measure the remaining standards.

d) Use this stored program number in the procedure above. Prepare a new calibration for each new lot of reagent, using the same stored program number.

SAMPLING AND STORAGE

Collect samples in clean glass or plastic bottles. Samples may be stored up to seven days by cooling to 4 °C (39 °F) or lower. Warm to room temperature before analysis.

ACCURACY CHECK

Standard Additions Method

a) Snap the neck off a Sulfate Voluette Ampule Standard Solution, 2500 mg/L.

b) Use the TenSette Pipet to add 0.1, 0.2, and 0.3 mL of standard to three 25-mL water samples. Mix each thoroughly. (For AccuVac ampuls, use 50-mL beakers.)

c) Analyze each sample as described above. The sulfate concentration should increase 10 mg/L for each 0.1 mL of standard added.

d) If these increases do not occur, see Standard Additions (Section I) for more information.

Standard Solution Method

Check the accuracy of the test by using the Sulfate Standard Solution, 50 mg/L, listed under Optional Reagents. Or, prepare this solution by pipetting 1.0 mL of the contents of a Voluette Ampule Standard for Sulfate into a 50-mL volumetric flask. Dilute to volume with deionized water.

PRECISION

In a single laboratory using a standard solution of 50 mg/L sulfate and two repesentative lots of powder pillows with the DR/2000, a single operator obtained a standard deviation of ± 0.9 mg/L sulfate.

In a single laboratory using a standard solution of 50 mg/L sulfate and two representative lots of AccuVac ampuls with the DR/2000, a single operator obtained a standard deviation of ± 2.2 mg/L sulfate.

INTERFERENCES

Silica and calcium may interfere at levels above 500 mg/L and 20,000 mg/L respectively.

Chloride and magnesium do not interfere at levels up to at least 40,000 and 10,000 respectively.

SUMMARY OF METHOD

Sulfate ions in the sample react with barium in SulfaVer 4 Sulfate Reagent and form insoluble barium sulfate turbidity. The amount of turbidity formed is proportional to the sulfate concentration. See *Chemical Procedures Explained,* Publication 7013, for more information.

REQUIRED REAGENTS (Using Powder Pillows)

Description	Quantity Required Per Test	Unit	Cat. No.
SulfaVer 4 Sulfate Reagent Powder Pillows	1 pillow	50/pkg	12065-66

REQUIRED REAGENTS (Using AccuVac Ampuls)

SulfaVer 4 Sulfate AccuVac Ampuls	1 ampul	25/pkg	25090-25

REQUIRED APPARATUS (Using Powder Pillows)

Clippers, for opening powder pillows	1	each	968-00

REQUIRED APPARATUS (Using AccuVac Ampuls)

Adapter, AccuVac Vial	1	each	43784-00
Brush	1	each	690-00
Vial, zeroing	1	each	21228-00

OPTIONAL REAGENTS

Sulfate Standard Solution, 50 mg/L	473 mL	2578-11
Sulfate Standard Solution, 1000 mg/L	473 mL	21757-11
Sulfate Standard Solution, Voluette ampule, 2500 mg/L, 10 mL	16/pkg	14252-10
Water, deionized	3.78 L	272-17

OPTIONAL APPARATUS

Beaker, 50 mL	each	500-41
Filter Paper, folded, 12.5 cm	100/pkg	1894-57
Flask, volumetric, 50 mL	each	14574-41
Funnel, poly, 65 mm	each	1083-67
Pipet, TenSette, 0.1 to 1.0 mL	each	19700-01
Pipet Tips, for 19700-01 TenSette Pipet	50/pkg	21856-96
Pipet, volumetric, 1.0 mL	each	14515-35
Pipet Filler, safety bulb	each	14651-00

For additional ordering information, see final section.
In the U.S.A. call 800-227-4224 to place an order.

Appendix D
Standard Operating Procedures

SOP-FL-010
AQUIFER HYDRAULIC TESTS

1.0 INTRODUCTION

This SOP provides the procedure for determining preliminary estimates of transmissivity and (T) storativity (S).

Once well development is complete, the existing airlift equipment, already downhole, can be used to test selected aquifer hydraulic characteristics. Preliminary estimates of transmissivity and storativity are useful in understanding aquifer characteristics and in designing longer-term pump tests.

- The well is airlifted for a specified period of time. Selection of the time period is based on review of the drilling and geophysical data for the respective hole, regional geologic and hydrologic data, and the probability of generating observable drawdown/recovery in the well. During the airlift portion of the test, the production rate is measured. Two methods are commonly used. Choosing which method depends on what discharge characteristics are encountered.

- If a constant flow is discharging from the well, a calibrated orifice or flow meter (connected to the discharge pipe) is the best method for flow measurement.

- Some wells discharge sporadically. Discharging fluid tends to evacuate the casing in discrete volumes as the 30 percent limit is approached, or if well inflow is limited. In this case, flexible polyethylene tubing (connected to the discharge pipe) absorbs the energy from the surging and totalizes the flow. A bucket and stopwatch are used to measure the flow rate.

- Upon completion of the airlift, water level measurements are taken. Any convenient measuring device (M-scope, wireline, chalk tape, pressure transducer, etc.) can be used. During this recovery period, the water level is rising. At the beginning of recovery, water level measurements are collected almost continuously because the water level is rising rapidly. As recovery slows, the time between measurements is lengthened because the recovery rate is slower. The period from when airlifting stops until one to two hours into recovery is most important. Accurate and quick measurements are essential. All data is recorded on the Aquifer Test Record.

- Measurement continues until the water level stabilizes (approximates the static water level before airlifting began).

- A time versus drawdown/recovery graph is plotted on semi-logarithmic graph paper. Time is plotted on the semi-log axis and recovery on the arithmetic axis. Plotting on semi-log paper yields a straight line graph.

- To calculate transmissivity, Jacob's equation (a modified form of the Theis Non-equilibrium equation) is used. Note: This is only a preliminary estimate of the aquifer's transmissivity. To calculate the slope of the recovery line, the end point water level height values over one full log cycle are determined. Thus, two heights are known, the smaller is subtracted from the larger, yielding a change (over period of time) (S) equal to the slope of the line.

- Transmissivity is then calculated using Jacob's equation.

$$T = \frac{264Q}{s}$$

Where: T = Transmissivity in gallons per day per foot (gpd/ft)
Q = pumping rate in gallons per minute (gpm)
s = slope of time-drawdown/recovery graph

(Note: With one well airlift tests only transmissivity is calculable. To calculate storativity, a two-well test is necessary. In this type of test, the water level in an observation well, spatially close to the airlifted well and completed in the same aquifer, is monitored during the airlift phase. Drawdown should occur in the observation well at some rate proportional to the production rate of the airlifted well. The drawdown is measured and plotted in the same fashion as the recovery data. However, the slope of the line in a drawdown test is decreasing as compared to the recovery test where it is increasing.)

- If a two-well test is performed, then use data obtained from measurements of the water level in the observation well. Calculate T using Jacob's equation, and the S once this is completed, use the equation (2.3 below) to generate a preliminary storativity value. (S = Storage coefficient)

- Also calculate a "T" and "S" based on the recovery data from the observation well. Compare the "S" and "T" values from drawdown and recovery.

2.0 SLUG TEST

Determine the I.D. of the piezometer casing. Measure the static water level in the hole with the transducer (the transducer may be calibrated while being lowered into the hole - see transducer operation). Calculate the volume (V_o) of water needed to produce the maximum head change without overflowing the casing ($V_o = h/r_c2$ where h = depth to water from top of casing; r_c = radius of casing interval where water level will be fluctuating). Round volume (V_o) down to the nearest easily measured volume (V). Use volume V to calculate H_o, the instant head change which will result from the injection of volume V ($H_o = V_o/r_c2$).

2.1 Instantaneous Recharge

The equations used to analyze a slug test assume an instantaneous recharge or discharge from the piezometer. Therefore, the "slug" of water should be introduced into the piezometer casing as rapidly as possible. Piezometers with water levels below 50 feet are better tested by methods described above.

Instantaneous recharge to the piezometer is approximated by rapidly pouring into the casing the volume V of water calculated in 2.0. The drop of the water level is then monitored closely by the transducer. If water cannot be poured into the casing without spilling, a funnel should be used to insure that the entire measured volume enters the piezometer. When all the water is in the casing, t = O. Water level readings should be taken in intervals as short as possible (no more than 5 second intervals at the beginning) and gradually increased as the rate of drop decreases. Monitoring should continue until the water level has stabilized. Values for H (where H equals the head inside the well at time t after injection or removal of the "slug", above or below initial head) are calculated by subtracting each water level reading from the initial Ho reading.

2.2 Suspended Weight

In piezometers where the static water level is deeper than 50 ft, the slug of water takes too long to travel down the casing for the recharge to even approximate instantaneous. These wells are better tested by introduction or withdrawal of a suspended weighted object of known volume. Water level changes are monitored as in 2.0. The suspended object should be large enough to displace sufficient water to cause an easily measurable change in head (H_o).

2.3 Calculations

Using the values of H, recorded at repeated intervals, values for H/Ho are computed and plotted on semilogarithmic paper. H/Ho is plotted on the linear axis of the paper and time, t, in seconds, on the logarithmic scale. Any convenient scale is acceptable for plotting H/Ho since this number is dimensionless. Plot the values and then curve match, superimposing the field value plot on plate 2 (see Reference), to define a match line for a value of t at $Tt/r_c2 = 1.0$ (match point values of H/Ho are not needed). Determine transmissivity, T, using:

$$T = 1.0r_c2/t$$

In addition, the storativity, S, is calculated using:

$$S = r_s2/r_c2$$

Where the value is determined through curve matching of the type curves (see Reference) and r_s = radius of screen zone

3.0 REFERENCES

S.W. Lohman. 1972. Ground-Water Hydraulics, Professional Paper 708. Washington, U.S. GPO.